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Technological Methods of Contemporary Processes in Refining Petroleum

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FDD doc No. 505128, Russian book, 1950, Moscow

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**UNCLASSIFIED - For Official Use Only****THE PROCESS OF THERMAL REFORMING**

The thermal-reforming process is used for the obtaining of high-octane automotive gasoline from low-octane ligroins.

The reforming stock (the ligroin), usually preheated to 75 - 80°C, is fed by pump 1, with a pressure at the discharge end not in excess of 60-70 atmospheres, into furnace 2, where the ligroin is heated to 540-550°C. Upon emerging from the furnace, the heated ligroin, after passing through a reduction valve, which reduces its pressure down to 8-12 atmospheres, enters a combined evaporator - fractionation column 4. Following fractionation, the nonstable reformed gasoline is drawn off from the top of the column, and, after cooling in coolers 5, is directed into collecting tank 8, from where the basic part enters into buffer tank 9. The spray from the collecting tank 8 is fed to the fractionation column 4.

From buffer tank 9, the nonstable gasoline, via the heat exchanger 12, is fed into stabilizer 10.

The hot side fraction from column 4 is utilized as the heat carrier for boiling pot 11, and then, after it is cooled, returns into the column.

A considerable part of the cooled-off heat carrier is preliminarily intermixed with the flow of products, emerging from the furnace, to attain the rapid cooling of the latter and to prevent the coking of the pipeline between the reduction valve 3 and the column 4.

The stable gasoline from the bottom of the column, after passing heat exchanger 12 and cooler 5, is drawn off into a plant storage tank.

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The reformed-residue from the bottom of column 4 follows into steam distillation column 6, from which, via cooler 5, it is pumped into a plant storage tank.

The nonstable liquid stabilizer distillate is drawn off into condenser-cooler 5, and then into tank 13. Part of this distillate is returned in the form of spray, and the excess - into a plant tank.

The upper distillate from the steam-distillation column 6 is condensed and cooled in cooler 5, and follows into collector 7. From this collecting tank, the driven-off distillate is fed into column 4 and to spray the steam-distillation column.

The balance sheet, and the characteristics of the initial materials [reforming-stock] and the products are given below.

Low-octane ligroins - to reforming	100%
Obtained:	
Reformed gasoline	80.0%
Gas	14.5%
Residue	5.5%

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Total	100.0%
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Consumed per 1 ton of refined raw material:

Fuel - in kilocalories	450-500
Steam under pressure of 10 atmospheres	125-180
Water - in cu meters	18 - 23
Electric Power - in kw-hours	2 - 3

Characteristics of Reforming Stock and Products Obtained

Reforming Stock:	
Specific gravity	0.7669

The reformed-residue from the bottom of column 4 follows into steam distillation column 6, from which, via cooler 5, it is pumped into a plant storage tank.

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Total	100.0%
-------	--------

Consumed per 1 ton of refined raw material:

Fuel - in kilocalories	450-500
Steam under pressure of 10 atmospheres	125-180
Water - in cu meters	18 - 23
Electric Power - in kwt-hours	2 - 3

Characteristics of Reforming Stock and Products Obtained

Reforming Stock:

Specific gravity	0.7669
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Characteristic factor	11.96
Distillation begins, at °C	98
10% distilled, up to °C	128
50% distilled, up to °C	157
90% distilled, up to °C	186
Distillation ends, at °C	198
Octane number	40.5

## Reformed Gasoline:

Specific gravity	0.7543
Reid vapor-pressure test, in millimeters of mercury column	517
Distillation begins, at °C	34
10% distilled-up to °C	63
50% distilled-up to °C	132
90% distilled - up to °C	182
Distillation ends - at °C	203
Octane number	70.2

## Residue:

Specific gravity	0.9452
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[Diagram] [Page 15 of original]

## Flowsheet for the Process of Thermal Reforming

1 - pumps; 2 - furnace; 3 - reduction valve; 4 - fractionation column; 5 - cooler; 6 - steam-distillation column; 7 - collecting tank; 9 - buffer tank; 10 - stabilizer; 11 - hot-water boiler; 12 - heat exchanger; 13 - tank for nonstable head.

## THE PROCESS OF THE COMBINED CRACKING OF THE LIQUID FRAC- TIONS AND GASES

This process is conducted at higher temperatures and pressures and is accompanied by greater transformation depth in the ligroins than during the usual thermal cracking or reforming.

Propane and butane, in diluting the stock to be cracked, which is ligroin, reduce the formation of coke and permit the increase in transformation depth. In addition, propane and butane, at high temperatures, are subject to cracking, with the formation of unsaturated hydrocarbons which, by undergoing polymerization, increase the gasoline yield.

The cracking stock (cold ligroin) is forced by pump 9 into absorber 8 for the absorption from the gases of propane and butane. Into the right bottom part of this absorber, the fraction propane-butane, in a liquid state, is fed from the side. The reduction of the temperature in the absorber is accomplished by the side flow, which is cooled in water-cooler 3.

The ligroin, saturated with the propane-butane fraction, is pumped via heat exchangers 10 into tubular furnace 1.

A pressure of 70-100 atmospheres is usually maintained in the coil of such a furnace, with the temperature of the product upon emerging from the furnace being 550-620°C.

Upon emerging from furnace 1, the flow of the hot product, in order to discontinue the reaction and prevent the formation of coke along the lines and in the apparatus, is intermixed with the product flowing from column 5 into hot-water boiler 6, steam-gener-

ator 12, and then follows into column 5.

The pressure in the combination column 5 is 20-26 atmospheres.

From the top of this column, the nonstable gasoline is drawn - it is condensated, cooled in water-cooler 3, and guided for stabilization into stabilizing column 7, from the bottom of which the stabilized gasoline is drawn into a storage tank.

From the top of column 7, the nonstable head (gas mixture) coming through condenser-cooler 3 into absorber 8, is drawn. The spray from the bottom left part of absorber 8 is pumped to the top of the stabilizing column. The condensate forming in condenser-cooler 3 is accumulated in the bottom left part of the absorber. A vertical partition in the bottom part of the absorber prevents the intermixing of the cracking stock with the spray of the stabilization column 7. A circulation spray is introduced into the upper and middle sections of the combination column of evaporator 5.

The residue from the bottom of the high-pressure column 5 is drawn into a low-pressure evaporator 2, in which, due to the drop in pressure, the wide fraction is distilled.

From the top of evaporator 2, the wide fraction is drawn, via cooler 3, into tank for distillate 4, from which tank the required part is returned in the form of a spray into the evaporator, while the surplus is drawn out of the installation.

From the bottom of evaporator 2, via immersion cooler 11, the heavy residue, which may be utilized as boiler fuel, is pumped out.

Below - see tabulated data on the cracking of light gas oil and of the wide gas oil fraction - with the addition of gas from the outside and without same.

SINGLE-STAGE CRACKING OF DIRECT -DISTILLATION GAS OIL BY WAY OF THE COMBINED CRACKING OF THE  
LIQUID FRACTIONS AND GASES

I N D E X E S	L i g h t g a s o i l			W i d e g a s o i l f r a c t i o n		
	Without the addition of gas from outside [2]	With the addition of propane-propane [3]	With the addition of butane-butene [4]	With the addition of gas from the outside [5]	With the addition of propane-propane [6]	With the addition of Standard butane [7]
[1]						
Content of nonsaturated hydrocarbons in the added gas - by volume	--	60	50	--	60	0
<u>Procedure:</u>						
Temperature upon emergine from furnace in °C	577	577	577	566	571	560
Pressure in furnace - in atmospheres	105	105	105	105	105	105
Dilution by gas, in % by volume of gasoil charged 20l		295	294	287	303	310
Amount of gas added from outside, in % by volume of gasoil charged	0	20	18	0	17	21
<u>Yield of products - in % by volume of gasoil charged:</u>						
Gasolien with vapor pressure, by Reid, of 517						
mm of mercury column	58.7	67.2	67.5	55.8	67.1	63.2

[Table continued]

[1]	[2]	[3]	[4]	[5]	[6]	[7]
Gas oil (20h - 315°C)	21.7	19.6	21.0	21.0	19.7	22.8
Mazut (petroleum residue) ( $\frac{7}{60}$ = 60)	11.9	13.7	13.8	18.6	17.4	20.5
<u>Characteristic of the cracking stock</u>						
Specific gravity	0.8468	0.8466	0.8534	0.8713	0.8618	0.8713
Aniline point - °C	76	76	78	77	77	77
Beginning of distillation - °C	216	216	217	141 <sup>1)</sup>	149 <sup>1)</sup>	141 <sup>1)</sup>
Distilled 10% - up to °C	258	258	266	263	269	263
Distilled 50% - up to °C	289	289	296	346	338	346
Distilled 90% - up to °C	332	332	365	461	412	461
End of distillation - °C	357	357	391	-	-	-
Characterizing factor	11.87	11.87	11.86	11.91	11.97	11.91
<u>Characteristic of gasoline:</u>						
Specific gravity	0.7385	0.7370	0.7416	0.7354	0.7313	0.7316
Bromine number	87	-	93	89	83	62
Aniline point - °C	25	29	23	27	29	32
Beginning of distillation - °C	41	37	41	39	33	38
Distilled 10% - up to °C	51	57	54	54	46	54

[Table cont'd]

[1]	[2]	[3]	[4]	[5]	[6]	[7]
Distilled 50% - up to °C	94	96	96	108	92	93
Distilled 90% - up to °C	176	169	181	187	180	171
End of distillation - °C	199	201	210	201	204	198
Octane number of the nonethylated gasoline	76.0	75.5	75.8	74.4	74.2	74.6
Octane number of gasoline to which 1.5 milliliters of tetraethyl lead fluid per liter was added	81.9	83.2	82.4	81.4	-	82.4
<u>Characteristic of the gas oil:</u>						
Specific gravity	0.9082	0.9260	0.9182	0.9088	0.9123	0.9088
Viscosity - <i>32</i>	1.12	1.15	1.18	1.15	1.18	1.15
Freezing point. - °C	-40	-40	-52	-37	-37	-34
Aniline point	27	-	28	26	20	32
Beginning of distillation - °C	203	229	243	216	236	225
Distilled 10% - up to °C	244	244	257	237	254	240
Distilled 50% - up to °C	259	273	270	259	271	266
Distilled 90% - up to °C	288	338	298	299	310	328
End of distillation - °C	306	363	321	314	337	328

[Table Cont'd]

[1]	[2]	[3]	[4]	[5]	[6]	[7]
<u>Characteristic of evaporator residue:</u>						
Specific gravity	1.038	-	1.032	1.036	1.036	1.032
Viscosity - $\eta_{30}$	58.5	-	56.1	57.3	49.6	51.2
Solidification point	2	-	-7	-1	-4	-1

1) Vacuum distillation

[Diagram, page 17 of original]

Flowsheet for the Process of the Combined Cracking of the Liquid Fractions and Gases

1 - tubular furnace; 2 - low-pressure evaporator; 3 - condenser-cooler; 4 - tank for distillate; 5 - combined column-evaporator; 6 - hot-water boiler; 7 - stabilization column; 8 - absorber; 9 - pumps; 10 - heat exchanger; 11 - immersion cooler.

THE PROCESS OF CONTINUOUS COKING OF THE HEAVY RESIDUES

One of the refining processes for the heavy nontransportable residues, with the simultaneous obtaining of gasoline, valuable distillates, and petroleum coke, is the process of continuous coking.

The residue stock is forced by pump 1 through the first two groups of heat exchangers 2, where it is preheated by the heat of the light and intermediate gas oils arriving from column 7 (in the flowsheet, for purposes of simplification, the lines of flow of the distillates and of the residue stock are not fully indicated).

Upon emerging from the two groups of heat exchangers 2, the residue stock is guided through a third group of heat exchangers 3, where it is preheated by the heat of the circulating reflux of column 4 (intermediate spray). Finally, in passing through the fourth group (by order of passage) of heat exchangers 2, the residue stock, preheated by the heat of the heavy gas oil, enters under the bottom plate of the main column.

The steam distillates from the coke chambers 12 enter the same column 4. As a result of rectification, the gasoline coking distillate is drawn from the top of the main column 4 and guided,



through cooler 5, into collecting tank 6, and from there, through gasoline heat exchanger 10, into stabilizer 9.

The stable gasoline is drawn from the bottom of the stabilizer into heat exchanger 10, cooler 5, and into storage tank, while the nonstable head is taken from the top of the stabilizer.

The light, intermediate, and heavy gas oils are taken from column 4 as side fractions. All these distillates are guided into the tri-sectional evaporator column 7, from where, following exaporation, they are forced by the respective pumps, through heat exchangers 2 and coolers 5, into storage tanks. The heavy residue from the main column 4 is forced by two centrifugal pumps, in separate flows, into two independent tubular furnaces 15. Just before entering the furnace, the basic flow is divided into four subsidiary flows, which pass through the convection section 16 of furnace, upon the emergence from which two flows proceed through the left-hand screen-grid section 15 of the furnace, while the other two flows proceed through the right-hand screen-grid section of the same furnace.

Upon emergence from the furnace, the flows of the left-hand and the right-hand sections become merged and enter the coking chambers 12. There are two intermittently operating coking chambers for each furnace. Each coking chamber of the described installation is an upright cylinder, five meters in diameter and 24 meters high.

Upon passing through the four-way fitting 13, the highly heated flow of the initial heavy residue, through the open stop valve 14 (the right-hand coking chamber is shown in the flowsheet as disconnected), enters the bottom part of chamber 12, and passes through its full height. Upon emerging from the coking chamber, the flow is

directed into the main column 4.

The highly heated product is left to linger for some time in the coking chamber, where it undergoes coking. The coke is gradually deposited in the chamber, while the coking products enter column 4.

When one of the chambers 12 is filled with coke, the flow from the furnace is directed into the other coking chamber, which is made ready to receive it. In order to disconnect the coking chamber that is filled, valves 14 are shut off, while valves 11 are opened up in order to connect the second chamber.

It is to be remembered that the removal of the coke from chambers 12 is done hydraulically (not indicated in flowsheet). To accomplish the above, the chamber is first air-blasted and cooled, then its bottom and top trap doors are opened, and a 450 mm hole drilled through the full height of the accumulated coke with the aid of a hydraulic drill. The hydraulic drill is attached to a hollow rod, which can be lowered and lifted with the aid of a pneumatic device installed above the coking chamber. Water is fed into the hollow rod under a pressure of 70 atmospheres.

After the drill has passed through the full height of the coke, it is removed through the bottom trap door, while a special hydraulic cutter, equipped with a series of jet terminals for guiding the water streams at various angles to the coke deposition in the chamber, is attached to the hollow rod.

The terminals of the upper part of the hydraulic cutter produce a horizontal water stream and cut off the coke in layers, while the terminals of the lower part of the hydraulic cutter pro-

duce a water stream at an angle to the top, as a result of which the coke becomes separated from the walls of the coke chamber.

The hydraulic cutter gradually moves upward. The coke together with the water falls downward, into a special car. The water is continuously led out into a settling tank, from where it is fed again by high-pressure pumps into the hydraulic system. The hydraulic method described reduces considerably the time required for the removal of the coke from the coking chamber, as well as the number of attending personnel, but, most importantly, no work of any kind is required in the interior of the coking chamber.

The process is characterized by the following indexes:

Mazut (petroleum residue) to coking	100.0%
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Obtained:

Gasoline	18.0%
Light gas oil	33.2%
Intermediate gas oil	15.8%
Coke	19.1%
Gas and losses	13.9%

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Total	100.0%
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Operating procedure of installation:

Ratio of circulating material to initial residue	0.33
Temperature on emerging from furnace	493°C
Temperature on entering coke chamber	482°C
Temperature on emerging from coke chamber	443°C
Pressure in coke chamber - in atmospheres	4.0

[Diagram, page 19 of original]

Flowsheet for the Process of Continuous Coking of Heavy Residues

1 - pumps; 2 - heat exchangers for the preheating of the initial residue by the heat of the distillates from evaporator column; 3 - heat exchangers for the preheating of the initial residue by the heat of the circulating reflux from main column; 4 - main column; 5 - coolers; 6 - collecting tanks; 7 - tri-sectional evaporator column; 8 - hot-water heater operating on the hot reflux; 9 - gasoline stabilization; 10 - gasoline heat exchanger; 11 - closed stop valves; 12 - coking chambers; 13 - four-way fitting for distributing product emerging from furnaces; 14 - open stop valves; 15 - screen-grid sections of tubular furnaces; 16 - convection sections of furnaces; 17 - saturated-steam inlet.

## DESTRUCTIVE DISTILLATION OF PETROLEUM-CATALYTIC PROCESSES

### PROCESS OF CATALYTIC CRACKING WITH MECHANICAL FEED OF THE CATALYST

This process is designated for the refining of gas oils and other heavier distillates for the obtaining of the basic component of aviation or automobile gasoline.

The catalyst used in the form of tablets or pellets of a 4 - 6 mm diameter is transported by a special elevator. The recent practice is to use mazuts [petroleum residues] of a direct distillation as the initial stock for the liquid-vapor phase feed of the reactor.

The residues to be cracked are fed by pump 26 into tubular furnace 25, upon emerging from which the heated stock is directed into evaporator 24 for the separation of the nonvaporized liquid residue.

The vapors of the initial residue to be cracked proceed from evaporator 24 into the upper part of reactor 22, where they come into contact with the hot catalyst arriving through uprise 5 from bin 6, and then undergo cracking.

The reaction products are drawn through line 23 into rectification column 3, while the poisoned catalyst descends from the reactor into the receiver of bucket elevator 7. The elevator lifts the poisoned catalyst into a bin 8 of regenerator 9. In the regenerator, the poisoned catalyst passes through a series of consecutive zones, in which the coke is burned out by blown-in air diluted with smoke gases coming from generator 16.

The heat contained in the smoke gases is utilized for the obtaining of water vapor, for which purpose water under pressure is circulated in pipes running inside the generator.

The regenerated catalyst, in descending, enters special buckets in elevator 7, which lifts it and empties it into feeder bin 6, From here, the catalyst, is continuously fed by gravity feed, via the uprise, into reactor 22.

It must be noted that the elevator buckets in up-to-date installations with a capacity of 700 tons per 24 hours, are divided into sections, of which some are picking up the poisoned catalyst, while the others pick up the regenerated catalyst.

Thus, one elevator simultaneously supplies the reactor 22 and the regenerator 9 with the respective catalyst.

Larger installations (above 700 tons per 24 hours) are usually equipped with two elevators having nonsectional buckets.

Gasoline and gases, as overhead products, are drawn from rectification column 3.

As side distillates of the rectification column, they draw the light gas oil, also the reflux product which passes through furnace 25 and, together with a fresh supply of cracking stock, is fed to catalytic cracking.

Part of the smoke gases from regenerator 9 are taken away by compressor 19 and fed into the upper part of the reactor 22 for the formation of a gas seal in the feeder uprise 5, while the remaining part of the smoke gases escape through the smokestack exhaust.

The fresh catalyst is fed into the system from feeder bin 12. Part of the regenerated catalyst is led off continuously into apparatus 11 for the purpose of blowing off the catalyst dust with the aid of the gas that is fed from generator 16. The catalyst dust, together with the gas enters the dust extractor 10, where it is precipitated. The precipitated dust from the dust extractor is fed into bin 13, from where it is periodically led out through line 14.

EXAMPLES OF OPERATION OF AN INSTALLATION WITH VAPOR-PHASE  
AND MIXED-PHASED FEED AND THE USE OF PELLET-SHAPED CATALYST

I n d e x e s	Vapor-phase feed (paraffin-base gas oil)		Mixed-phase feed (Heavy gas oil)
	Case I	Case II	
[1]	[2]	[3]	[4]
Activity index of catalyst	36.6	35.9	35.6
Fresh Stock to cracking, cu meters/24 hrs	1451	1255	1520
Including stock in liquid phase, cu meters/24 hours	-	-	293
Circulating stock, cu meters/24 hrs	910	668	-
Catalyst circulation -tons/hour	108	122	120
Vapor temperature when entering reactor - °C	463	457	471
Catalyst/temperature when entering re- actor - °C	513	482	519



[1]	[2]	[3]	[4]
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Yield of Products Predicated on Fresh Stock:

Debutanized gasoline (90% up to 190°C),

% by volume	52.7	50.5	42.6
Gas oil - % by volume	32.4	32.9	50.1
Cytt <sub>8</sub> - % by volume	3.9	3.9	2.7
Iso - Cytt <sub>10</sub> - % by volume	7.6	8.5	4.7
Cytt <sub>10</sub> - % by volume	3.2	3.0	2.0
Dry gas - % by volume	6.2	5.8	3.9
Coke - % by volume	3.5	5.3	3.0

Vapor	Liquid
phase	phase

[1]	[2]	[3]	[4]	[5]
-----	-----	-----	-----	-----

Characteristic of fresh cracking stock:

Specific gravity	0.8545	0.8560	0.8905	0.9291
Beginning of distillation - °C	202	282	244	291
Distilled 90% - up to °C	356	372	436	-
Sulfur content - % by weight	0.32	0.29	0.31	-
Coke, as per Konradsen, % by weight	0.02	-	0.01	-

Characteristic of gasoline with a Reidvapor pressure of 51.7 mm of mercurycolumn:

[1]	[2]	[3]	[4]
Octane number of the nonethylated gasoline	80.0	79.3	79.8
Octane number of gasoline, to which 1.5 milliliter of lead tetra-ethyl fluid per 1 liter of gasoline was added	87.9	88.2	87.1
Sulfur content, % by weight	0.017	-	0.041



[Diagram, page 21. of original]

#### Flowsheet for the Process of Catalytic Cracking with Mechanical Feed of

##### Catalyst

1- gas separator; 2 - condenser - cooler; 3 - rectification column;  
4 - hot-catalyst depository; 5 - uprise for the descent of the catalyst; 6 - feeder bin; 7 - elevator; 8 - regenerator bin; 9 - regenerator; 10 - cyclone dust separator; 11 - catalyst-dust blowoff apparatus;  
12- fresh-catalyst bin; 13 - dust bin; 14 - line of descent for the catalyst dust; 15 - air blower; 16 - smoke - gas generator; 17 - vapor separator;  
18 - vapor - water mixture feed line; 19 - turbo-compressor; 20 - line of descent for poisoned catalyst; 21 - distribution grids for catalyst;  
22 - reactor; 23 - feed line for reaction products; 24 - evaporator;  
25 - tubular furnace; 26 - pump.

#### THE PROCESS OF CATALYTIC CRACKING WITH PNEUMATIC FEED OF

##### CATALYST

The process is designated for the refining of gas oil with the object of obtaining the basic component of high-octane aviation gasoline or automobile gasoline having an octane number of 80-82. (For the obtaining of basic component of aviation gasoline, a two-stage catalytic-cracking process is employed. The gasoline (automobile) obtained from the stock in the first stage (cracking) is subject to a repeat catalytic treatment (purification). The yield of the basic aviation-gasoline component from the initial stock is approximately one half of the yield of the automobile gasoline obtained by single stage cracking).

As the initial stock, any distillate, and even deasphalted mazut may be used. The catalyst is usually ground to pass through

a 300-mesh screen.

The cracking stock is forced by pump 13 through tubular furnace 12 (where it is vaporized), and from there into reactor 7. Ahead of the reactor, to this flow of the cracking stock is adjoined the flow of the regenerated pulverulent catalyst, which is descending from the regenerator along the uprise 5, its temperature being 530-620°C. The flow of the hot cracking stock, having become intermixed with the flow of the hot catalyst, enters the lower part of reactor 7.

The reaction products, coming out from the reactor, follow into rectification column 8.

The vapors emerging from reactor 7 carry with them the pulverulent catalyst, which is partially intercepted by the cyclone dust separators 3. The remaining part of the catalyst is precipitated at the bottom of rectification column 8 and is continuously returned together with the diluent (heavy gas oil) into reactor 7.

To restore the activity of the catalyst, the latter is continuously drawn from the reactor into column 6, where it is steam-distilled. The steam-distilled catalyst descends, by way of uprise 5 to the collector, into which is simultaneously fed compressed air from compressor 11. The coke is burned out from the catalyst in regenerator 4 by the compressed air preliminarily diluted by the smoke gases.

The regenerated catalyst is drawn from regenerator 4, via-uprise 5, to another collector, into which is also fed the fresh cracking stock from the tubular furnace.

The smoke gases formed as a result of the coke combustion,

for the purpose of intercepting the catalyst, are directed, via the cyclone dust separator 3, into the tubular cooler 2, where they are cooled by water with the simultaneous formation of vapor, and, after passing electric filter 1, they are exhausted into the open air. The amount of catalyst precipitated by the electric filter is returned to regenerator 4. The reduction in the loss of catalyst is of exceptional importance, since the cost of catalyst is very high.

As an example of one of the operational versions of such an installation, the procedure, material balance sheet, and the qualitative indexes of the cracking stock and the products obtained, when operating on heavy stock with rectification, are cited below. The catalyst used in this case is a mixture of synthetic and natural catalyst, with the latter predominant.

<u>To cracking:</u>	[1]	[2]
Fresh stock, cu meters/24 hours -		2808
Reflux material, cu meters/24 hours -		364

<u>Obtained:</u>	
Dry gas - % by weight	7.5
Propane-Propene - % by volume -	3.8
Butane-butene - % by volume -	8.2
Light gasoline - % by volume	18.1
Heavy gasoline - % by volume	17.1
Light gas oil - % by volume	20.9
Heavy gas oil - % by volume	26.9
Coke - % by weight	7.9

<u>Consumed per 1 ton of stock:</u>	
Steam under pressure of 10.5 atm (absolute) in kg for pumps	118

[1]	[2]
for steam-distillation -	40
<hr/>	
Total	158

Obtained from the Economizer Boiler:

Steam for fractional-distillation reflux - in kilograms	217
Excess steam, in kilograms -	59
Electric energy for power and lighting, in Kilowatt-hours -	1.3
Fuel valve, in kilo-calories, for compressors -	176
Water at no higher than 30°C, in cu meters	10

Procedure when Installation Operates on Heavy Cracking Stock:

Temperature in reactor - in °C	509
Temperature in regenerator - in °C	596
Ratio of catalyst to stock -	10.3
Pressure in reactor - in atmospheres (excess)	0.64
Pressure in regenerator, in atmospheres (excess)	0.07

## CHARACTERISTIC OF CRACKING STOCK AND PRODUCTS OBTAINED

Physical and Chemical Properties	Cracking	Light	Heavy
	Stock	Gasoline	Gasoline
[1]	[2]	[3]	[4]
Specific gravity	0.9165	0.7121	0.8280
Beginning of distillation - °C	249	42	131
10% distilled - up to °C	348	54	141
50% distilled - up to °C	424	72	163
90% distilled - up to °C	501	103	200
Sulfur content, % by weight	0.4	0.1	0.2
Aniline point, °C	74	22	4
Coke - as per Konradsen	0.2	-	-
Octane number - by the motor method	-	79	79

Note: The above consumption indexes pertain to an installation without a tubular furnace. The preheating of the stock to the cracking temperature is accomplished by the utilization of the heat of the hot distillates and of the regenerated catalyst. The indexes tabulated above are characteristic of the work of the installation for the production of automobile gasoline.

[Diagram, page 23 of original]

Flowsheet for the process of catalytic cracking with pneumatic feed  
of catalyst

1- electric-precipitation filter; 2- cooler for smoke gases; 3 - cyclone dust separator; 4 - regenerator; 5 - uprisers for the descending of the catalyst; 6 - column for the steam-distillation of

the poisoned catalyst; 7 - reactor; 8 - rectification column; 9 - condensor-cooler; 10 - water-gas separator; 11 - compressor; 12 - tubular furnace; 13 - pump.

#### THE PROCESS OF CATALYTIC CRACKING OF THE STOCK WITH CATALYST IN SUSPENSION

The distinguishing feature of this process is the use of relatively small amounts of catalyst.

As the inexpensive and low-activity catalyst, they frequently utilize clay that was used for the contact purification of lubricating oils. However, finely-ground natural and synthetic catalysts can also be used.

The preparation of the catalyst paste is accomplished in the following manner.

From bin 6, the catalyst (clay) enters into mixer 5, into which is simultaneously forced by pump 8 a diluent from tank 9 for intermixing with the catalyst. The intermixing is accomplished with the aid of a propeller mechanism.

The formed paste is fed by pump 4 into the line, along which the cracking stock is proceeding after having passed through heat exchangers 11. The resultant mixture passes in two flows through convection section 3, radiation section 2, and, along line 1, enters the combination column 12.

Into the two flows of the hot cracking stock is pumped from the tubular furnace the cooled gas oil, which is drawn off from column 12. The gas oil passes through heat exchanger 11, immersion

cooler 10, and is forced by a pump into the two flows emerging from the furnace prior to their entering the combination column 12.

In the combination column, the vapors are washed to remove the catalyst intercepted by them before, and the petroleum products undergo rectification.

From the top of column 12, they draw the gasoline, which is condensated and cooled in cooler 13, then enters gas separator 15, and from there -- to the plant storage tank.

Into the upper section of the column, along line 14, the ligroin-kerosene spray is drawn. Into the middle part of the column (into several points of its height), for the purpose of washing the vapors proceeding along line 16, the gas oil spray is fed. Simultaneously, part of the gas oil from column 12 enters into steam-distillation column 17, from where it proceeds, via cooler 13, into a plant storage tank, and is partly returned into tank 9. From the bottom of the column, the residue, with the clay (the catalyst) in suspension, enters via cooler 10, into collecting tank 21, and via line 20 - to rotary filter 19.

The filtered cracked residue enters tank 22, from where it is pumped into a plant storage tank.

The catalyst (clay), depending on its value, can be sent either to regeneration or to the waste dump.

The process just described is suitable for the production of automobile gasoline with a stepped-up octane number. The presence of a pulverulent catalyst in the cracking-stock flow, proceeding through the tubular furnace, and the high temperature in the latter

call for the use of pipes made from alloy steel and return bends of stepped-up strength - in order to avoid rapid wear of the latter. The most complex part of the installation is the section for the continuous filtration of the cracked residues, for which hot filters of special construction are required.

The indexes for the cracking of gas oil, with the use of natural and synthetic catalysts, are given in Table below.

THE CRACKING OF GAS OIL WITH THE USE OF NATURAL AND  
SYNTHETIC CATALYST

Indexes	Clay used Fresh Synthetic		
	up in the clay catalyst		
	purifica-		
	tion of		
	lubricants		
[1]	[2]	[3]	[4]
Pressure on emerging from furnace,			
in atmospheres (excess)	25	25	25
Temperature on emerging from furnace,			
in °C	568	574	568
Catalyst consumed kg/M <sup>3</sup>	-	-	28.5
<u>Yield of Products:</u>			
Stable gasoline - in % by volume	-	-	52.4
Excess butane - in % by volume	-	-	4.6
Distillation fuel (212-315°C)			
- in % by volume	-	-	18.8
Mazut (above 315°C), in % by volume	-	-	12.2
Dry gas - in % by volume	-	-	15.3
Octane number of nonethylated			
gasoline	73.2	73.5	75.4



[Diagram, page 25 of original]

Flowsheet for the Process of Catalytic Cracking of Stock  
with Catalyst in Suspension

1 - emergence of products from furnace; 2 - multichamber radiation furnace; 3 - convection section of furnace; 4 - pump for feeding catalyst paste; 5 - catalyst-paste mixer; 6 - catalyst bin; 7 - catalyst inlet; 8 - pumps; 9 - tank for the paste diluent; 10 - immersion coolers; 11 - heat exchangers; 12 - combination column; 13 - tubular coolers; 14 - line for spraying the column with the ligroin-kerosene fraction; 15 - gas separator; 16 - line of the gas oil spray; 17 - steam-distillation column for gas oil; 18 - inlet for the open water vapor; 19 - rotary filter; 20 - residue feed line; 21 - residue-collecting tank; 22 - filtrate- receiving tank.

THE PROCESS OF CATALYTIC REFORMING IN THE PRESENCE OF  
HYDROGEN (HYDROFORMING)

One of the up-to-date catalytic processes for the simultaneous obtaining of a high-quality aromatic component of aviation fuel and toluene is the hydroforming process, i.e. the process of catalytic reforming in the presence of hydrogen. In hydroforming, the catalyst is located in the reactor in a fixed position, where it is periodically subject to regeneration.

For the continuity of the process, the installation usually contains two pairs of reactors, of which one pair is within the reaction cycle (feeding of the stock), while the other pair is within the regeneration cycle (burn-out).

The crude stock (ligroin\* is forced by pump 11, via heat-

exchanger 13, into one of the coils of tubular furnace 14, where it is heated to 510°C. From furnace 14, the heated stock is fed into the reactor of first stage 1. Simultaneously, via the other coil of furnace 14, compressor 12 feeds the circulating gas with a considerable hydrogen content. The gas is heated in the furnace to 540-560°C. The two flows (the ligroin and the gas) are intermixed at the entrance to the reactor of the first stage 1. Upon emerging from the reactor, the common flow is directed into a separate coil of the tubular furnace 14, where the products are reheated and fed into the reactor of the second stage 5. From reactor 5, the hot products pass a number of heat exchangers 13, where they are somewhat cooled. On emerging from the heat exchangers, the products are fed into water cooler 9 and gas separator 10, after which they are forced by a pump to rectification.

Part of the gas from gas separator 10 is drawn into an absorber, while the remaining part is driven by compressor 12, via heat exchanger 13 and tubular furnace 14, into the reactor of first stage 1. At the same time, the second pair of reactors, for the purpose of regenerating the activity of the poisoned catalyst, are burning through, the air fed by compression 2, after being diluted by the smoke gases flowing from turbocompressor 3, is directed (from the top down) into reactor 4, in which the burning-out of the coke from the catalyst takes place.

Part of the obtained smoke gases is returned to the intake of compressor 3, while the other part follows into reactor 8 (from the bottom upward) for the preheating of the catalyst to the required temperature of approximately 510°C, ahead of its next inclusion into the reaction cycle.

The smoke gases follow from reactor 8 into waste-gas boiler 6, where their heat content is utilized for the formation of water vapor, which flows into vapor-collector 7, and is fed from there to respective technological requirements. The cooled smoke gases are exhausted through the smokestack

#### OPERATING SCHEDULE AND YIELDS

##### Yield of Products - on the Basis of Stock:

Gas - % by weight	8.5
Coke on catalyst - % by weight	1.3
Aviation-gasoline component - % by volume	76.0
Polymers heavier than gasoline, % by volume	9.3

##### Consumed per 1 Ton of Cracked Stock:

Fuel, in kilograms	285
Steam, in kilograms	273
Water, in cu meters	33
Electric power, in kilowatt-hours	58

##### Operating Schedule:

Volumetric rate per hour	0.65
Duration of reaction cycle, in hours	4.2
Temperature of flow on contacting the catalyst	
- in °C.	527
Temperature of flow reduced in passing the catalyst, by °C	63
Mean temperature of catalyst, °C	471
Pressure in the reactor, in atmospheres (excess)	14.6
Ratio of circulating gas to stock, M <sup>3</sup> /M <sup>3</sup>	1210

## THE CHARACTERISTIC OF THE CRACKING STOCK AND THE GASOLINE

Physical and Chemical Properties	Cracking Stock	Aviation Gasoline Component
[1]	[2]	[3]
Specific gravity	0.7724	0.7927
Aromatic Compounds , % by volume	10	44
Bromine number, in g/100g	9	3.1
Octane number - with the addition of 2.6 milliliters of tetraethyl lead fluid per 1 liter of gasoline	-	93.7
Beginning of distillation, °C	97	57
10% distilled, up to °C	111	90
50% distilled, up to °C	126	119
90% distilled, up to °C	146	141
End of distillation, °C	166	166

[Diagram, page 27 or original]

Flowsheet for the Process of Catalytic Reforming in the Presence of Hydrogen (Hydroforming)

1 - Reactor of the first stage; 2 - air compressor; 3 - turbocompressor for smoke gases; 4 - reactor in the phase of burning-out (from top downward); 5 - reactor of the second stage; 6 - waste-gas boiler; 7 - steam collector; 8 - reactor in the phase of catalyst preheating (from the bottom upward); 9 - cooler; 10 - gas separator; 11 - pumps; 12 - circulating-gas compressor; 13 - heat exchangers; 14 - combination multicoil furnace.

## THE PROCESS OF OBTAINING XYLIDINE BY HYDROGENERATION UNDER HIGH PRESSURE

The crude stock for the production of xylidine by hydrogenation is mononitroxylene. Hydrogeneration is conducted under high pressure, with the use of molybdenum sulfide on activated carbon as the catalyst. In order to sustain the activity of the catalyst, hydrogen sulfide is introduced into the system, in addition to the initial stock.

The reaction of hydrogenation of nitroxylene is accompanied by a considerable liberation of heat. Hence, in order to control the temperature cycle, water and the cooling gas, which circulates in the system, are admitted into the reactor.

The nitroxylene is fed by pump 2 into absorber 3, where it becomes saturated with hydrogen sulfide delivered by compressor 1. From the bottom of absorber 3, the nitroxylene flows toward reactor 5, before entering which it is intermixed with the flow of the cooling gas and of the circulating mixture of the raw xylidine and water.

The products of the reaction follow from the reactor, via heat exchanger 6 and cooler 7, into high-pressure gas separator 8. The separated gas then returns to reactor 5, while the raw xylidine is transferred into a low-pressure separator 9.

The gas separated in the latter is driven out of the installation, while the xylidine follows into settling tank 10, from where, after the water is separated from it, it is drawn into a storage tank.

Part of the xylidine and part of the water in settling tank 10 are pumped by separate pumps, become intermixed in the

common pipeline, and enter the suction of the pump, which is returning the circulating mixture of water and xylidine into reactor 5. This mixture, ahead of its entry into reactor, becomes heated in heat exchanger 6 by the heat of the products, coming out of the reactor, and is additionally heated in preheater 4.

Hydrogen is fed into the system by compressor 1 and is mixed in the pipeline with the circulating gas.

For the control of the reaction temperature, a vapor condensate is fed into the suction of the circulating pump, which returns the raw xylidine into the reactor.

The return of the raw xylidine mixed with water (one part of xylidine to one part of water) helps preserve the activity of the catalyst.

To prevent the accumulation of inert components in the circulating gas, part of the high-pressure gas is continuously eliminated from the installation.

For the obtaining of a commercial product, the raw xylidine is redistilled, with the elimination of the diamino compounds obtained during the reduction of dinitroxylenes present in the raw stock sent to hydrogenation.

Commercial xylidine is used as a component of aviation gasoline - it increases the anti-knocking stability of the latter in rich mixtures. The usual addition of xylidine amounts to 5 percent.

**TYPICAL INDEXES FOR THE OBTAINING OF XYLIDINE BY THE  
HYDROGENATION OF NITROXYLENES UNDER HIGH PRESSURE,  
WITH MOLYBDENUM SULFIDE ON ACTIVATED CARBON USED AS  
A CATALYST**

Indexes	Without xylidine circulation [2]	With xylid- ine circu- lation [3]
[1] Nitroxylene, volume/volume of catalyst/hour	0.44	0.44
Circulation of raw xylidine, volume/volume of catalyst/hour	0	0.4
Circulation of water, volume/volume catalyst/hour	1.6	1.2
Pressure in the system, in atm.	210	210
Temperature on entering reactor, °C	165	168
Maximum temperature in reactor, °C	224	224
Circulating and cooling gas, m <sup>3</sup> /m <sup>3</sup> of total stock (water + nitroxy- lenes + circulating xylidine)	1425	1425
Hydrogen content in circulating and cooling gas, % by volume	80	85
Hydrogen sulfide content in circu- lating and cooling gas, % by volume	0.6	0.6
Conversion of nitroxylene, %		
<u>Properties of the Crude Stock and The Products:</u>	Crude Stock [2]	Pro- ducts [3]
[1]		Crude Stock [4]
Specific gravity at 15.5/15.5 °C	1.116	Pro- ducts [5] 1.112 0.971

[1]	[2]	[3]	[4]	[5]
Xylidine content, % by weight	0	85.6	0	88.2
<u>Nitroxylenes content, % by weight</u>				
Mononitroxylene	94.6	0.6	93.4	0.1
Dinitroxylene	1.8		1.6	
Water content, % by weight	Traces	2	0.2	2

[Diagram, page 29 of original]

Flowsheet for the Process of Obtaining Xylidine by Hydro-  
genation under high pressure

1 - compressors; 2 - pumps; 3 - absorber; 4 - preheater; 5 - re-  
actor; 6 - heat exchanger; 7 - cooler; 8 - high-pressure separator;  
9 - low-pressure separator; 10 - settling tank.



### THE REFINING OF GASES

#### THE PROCESS OF THE SELECTIVE ADSORPTION OF HYDROCARBONS FROM GASES UNDER PRESSURE

The process reduces itself to the selective extraction of hydrocarbons from gases by the method of adsorption with activated carbon under pressure. The selective capacity depends on the adsorption temperature and the molecular weight of the hydrocarbons.

Below follows the description of the process of the adsorption of ethene from a gas mixture.

The initial stock (gas mixture) having a temperature of 60°C enters into the middle part of adsorber 6 and flows upward to encounter the descending activated carbon, with which it comes into close contact. The carbon absorbs the ethene and other gases having the same or a higher molecular weight. The gas mixture, deprived of the ethene and consisting basically of methane and hydrogen, rises upward and, at the cooling sector, is led out into cyclone dust separator 15 and from there into the fuel network.

The adsorbed gases, primarily ethene, together with the carbon adsorbent, are led downward and enter heating sector 10. In this sector, the temperature of the adsorbent is sustained at a level of 265°C. The heat is imparted with the aid of a circulating heat carrier. Due to high temperature, the adsorbed ethene is completely separated, and follows, via line 8 into dust separator 15, water-vapor condenser 13, drier 14 (where the water is separated), and then into storage tank.

Free water vapor is admitted into the bottom part of adsorber 6, where the adsorbent, acting as a shut-off device, prevents the

water vapor from penetrating into the uprise of the pneumatic-flow pipe 3.

From the bottom of adsorber 6, the carbon mass is entrapped and carried upward by the blow-off gas fed from the gas blower 16, while its surplus is fed into the fuel network.

For sustaining the activity of the carbon, the installation is equipped with tubular carbon activator 22, into which flows part of the carbon from the top of adsorber 6. The carbon mass entering through the tubes is heated and undergoes live-steam distillation, basically for the purpose of eliminating the heavy hydrocarbons, which interfere with the activity of the carbon.

The carbon in the activator is heated by the smoke gases flowing from combustion chamber 21. The smoke gases pass along the space between the pipes and are exhausted through the smokestack.

The activated carbon in use is made up of particles that pass through a 12 - 30 mesh screen.

The rate of flow of the carbon in the adsorber is controlled by a special vibrator plate 12 located directly under the heating section 10. The vibration mechanism consists of two stationary plates 26 and 27 and one movable plate 12 in the middle. The clearances between the plates are about 6 millimeters, which practically excludes the possibility of attrition for the carbon.

The movable vibration plate has a cluster of interfluent tubes which serve simultaneously for the admission distribution, and passing of the descending flow of the carbon adsorbent. When the movable vibration plate 12 is in its extreme position the carbon

is fed from plate 26 onto plate 12 and is somewhat detained in the above mentioned tubes, since the perforations in stationary plate 27 do not coincide with the openings of the interfluent tubes of the movable vibration plate 12. When vibration plate 12 moves into its opposite extreme position, the openings of the interfluent tubes, which are filled with carbon, will at some moment coincide with the perforations of the bottom stationary plate 27, and the carbon from the tubes will pass downward through the perforations of this plate.

The vibratory plate is driven by piston 28 tied in with a hydraulic-system drive.

THE MATERIAL-BALANCE SHEET AND THE COMPOSITIONS OF THE GAS FLOWS

Components	Gas Mixture		Target gas		Exhaust Gas		Blow-off gas	
	to adsorption							
	m <sup>3</sup> /hour	% by volume	m <sup>3</sup> /hour	% by volume	m <sup>3</sup> /hour	% by volume	m <sup>3</sup> /hour	% by volume
[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]
Hydrogen	832.5	39.8	-	-	402.0	31.6	430.4	61.8
Nitrogen	35.1	1.7	-	-	17.8	1.4	17.3	2.5
Carbon Monoxide	18.9	0.9	-	-	9.9	0.8	9.1	1.3
Oxygen	2.8	0.1	0.1	0.1	1.3	0.1	1.4	0.2
Methane	1073.2	51.3	-	-	838.1	66.1	235.0	33.7
Carbon dioxide	4.2	0.2	3.7	2.9	-	-	0.6	0.1
Acetylene	4.5	0.2	4.5	3.6	-	-	-	-
Ethene	120.3	5.8	117.5	92.7	-	-	2.8	0.4
Ethane	0.9	Traces	0.9	0.7	-	-	-	-
TOTAL	2092.4	100.0	126.7	100.0	1269.1	100.0	696.6	100.0

Consumed when passing a gas mixture of 2125 m<sup>3</sup>/hour:

Water, in cu meters	43.6
Fuel (gas), in cu meters	83.5
Steam under a pressure of 10.5 atmospheres, in kilograms	616.8
Electric power, in kilowatts	11.0
Activated carbon, in tons/hour	8.2

[Diagram, page 31 of original]

Flowsheet for the Process of Selective Adsorption of  
Hydrocarbons from Gases

Under Pressure

1 - bin for activated carbon; 2 - line of excess carbon discard;  
3 - uprise of pneumatic-flow pipe (lift) 4 - cooling section;  
5 - water inlet; 6 - adsorber; 7 - gas-distribution plates; 8 -  
ethene discharge; 9 - heat-carrier inlet; 10- heating section  
with circulating heat carrier; 11- heat-carrier discharge; 12 -  
vibratory plate with interfluent tubes; 13- condenser and cooler  
for steam; 14 - drier; 15 - cyclone dust separators; 16 - gas  
blower; 17 - gas-blower feedline to pneumatic-flow pipe; 18 -  
line for the addition of activated carbon; 19 - air blower; 20 -  
shut-off control device; 21 - smoke gas generator; 22 - tubular acti-  
vator for carbon; 23 - operational-liquid inlet; 24 - operational-  
liquid discharge; 25 - liquid distributor; 26 - stationary plate  
with tubes and caps; 27 - stationary plate with perforations;  
28 - piston; 29 - switch.

THE PROCESS OF PHOSPHORIC-ACID TOTAL POLYMERIZATION

This process is used for the catalytic polymerization of  
nonsaturated gases for the purpose of obtaining high-octane poly-

merized gasoline for automobiles.

These installations very frequently operate in combination with cracking plants for the refining of the nonsaturated gases from the cracking process.

The initial stock (gas) is fed by compressor 1 into tube heater 4, where it is heated to 200°C. The heated stock enters consecutively into catalyst chambers 5 filled with phosphoric-acid catalyst tablets.

Simultaneously, a special water injection pump 3 feeds water from measuring tank 2 into the flowline of the stock ahead of the furnace and into the flowline of the stock following its emergence from the first (in the order of the flow of the stock)catalyst chamber. Thus, the initial stock enters the catalyst chambers together with the water vapor.

Under the effect of the phosphoric acid, the non-saturated hydrocarbons undergo polymerization, with the formation of polymerized gasoline.

Upon emerging from the second (in the order of flow of the stock\* catalyst chamber, the polymerized-gasoline vapors are partly condensated in water condenser and, with a temperature of 175°C, enter stabilizer 8, which is equipped with a casing head.

In the upper part of the stabilizer, there is a water condenser 7, which provides for the spraying of the stabilizer and cools the waste gas. Part of the latter, together with a fresh supply of gas, enters furnace 4.

The circulating gas controls the percentage of nonsaturated hydrocarbons in the stock mixture, since an excessive content of

hydrocarbons in the latter may result in the excessive formation of high-boiling polymers.

From the bottom of the stabilizer, to polymerized gasoline is directed into hot-water kettle 9, and from there, via water-cooler 10, into storage tanks.

The following degree of conversion of the olefins is attained in the process of phosphoric-acid polymerization: ethene -- up to 40 percent, propenes -- up to 98 percent, butenes -- up to 92 percent, and isobutenes -- up to 94 percent. The yield of the polymer-distillate is up to 800 liters per 1000 cubic meters of initial stock.

The indexes, which are characteristic of the properties of the polymer-distillates obtained, and the consumption indexes are depicted in the Table below:

#### CHARACTERISTICS OF THE POLYMER-DISTILLATES

Indexes	INITIAL STOCK		
	Stabilized cracked- gasoline head	Butane- butene fraction	Propane- propene fraction
[1]	[2]	[3]	[4]
<u>Operating Procedure:</u>			
Pressure, in atmospheres (excess)	35	70	70
Temperature, in °C	185-188	204	232

[Table Cont'd]

[1]	[2]	[3]	[4]
<u>Properties of the Polymer-Distillates:</u>			
Specific gravity	0.7238	0.7227	0.7298
Beginning of distillation, °C	36	26	62
10% distilled, up to °C	69	67	96
50% distilled, up to °C	113	126	130
90% distilled, up to °C	186	193	166
End of distillation, °C	221	213	206
Reid vapor pressure, in mm of mercury column	465	569	207
Octane number -- without the ad- dition of tetraethyl lead fluid	-	82.5	-
Octane number -- with the addition of 1.5 milliliters of tetraethyl lead fluid per liter	-	85.0	-
Octane number of mixture 5 - 10 per- cent in a mixture with direct- distillation gasoline	-	-	110-120
Octane number of mixture 5 - 10 per- cent in a mixture with gasoline from thermal cracking	-	-	95-100
Consumed per 1 ton of polymer-distillate:			
Steam, in kilograms		2360	
Water, in cubic meters		118	
Electric power, in kilowatt-hours		31	

[Diagram, see page 33 of original]

Flowsheet for the process of phosphoric-acid total polymerization

1 - compressor; 2 - water tanks; 3 - water-injection pump; 4 - tube heater; 5 - catalyst chamber; 6 - condenser; 7 - water condenser; 8 - stabilizer with casing head; 9 - hot-water kettle; 10 - water cooler.

THE PROCESS OF SULFURIC-ACID ALKYLATION OF ISOBUTANE WITH THE AID OF BUTENES

The process of alkylation of isobutane with the aid of butenes, in the presence of concentrated sulfuric acid, permits the obtaining of a high-octane aviation gasoline component (technical isooctane). The essence of the process is as follows.

The initial stock (the butane-butene fraction) is passed through alkali mixer 1, where it is treated by a circulating alkaline solution for the elimination of hydrogen sulfide, and then drawn into settling tank 2.

From the settling tank, the butane-butene fraction, having intermixed preliminarily with the circulating isobutane, follows into heat exchanger 9, ammonia cooler 10, and enters the contactor (reactor) 5.

Into reactor 5, along line 4, flows a concentrated sulfuric acid, which, with the aid of mechanism 7, is thoroughly intermixed with the components to be alkylated at a temperature  $0^{\circ} - 10^{\circ}\text{C}$ . For the purpose of cooling the reaction masses, ammonia is circulated in special tubes of contractor 5. The products of alkylation follow



along line 6 into sulfuric-acid settling tank 8, from where the settled out sulfuric acid is returned by pump 3 into contactor 5, while the used-up sulfuric acid is led out of the system.

From settling tank 8, the products of alkylation pass through heat exchanger 9, alkali mixer 1, and, after treatment by the circulating alkaline solution, follow into alkali settling tank 2.

From settling tank 2, they alkylation products follow, via preheater 15, into de-isobutanizer 14.

The isobutane, which did not participate in the reaction, together with the propane, are drawn from the top of the de-isobutanizer 14, cooled in condenser 13, then follow into collecting tank 17 and storage tank 18, from where part of the flow, via heat exchanger 9, is drawn into de-propanizer 19 for the elimination of propane, in order to avoid the accumulation of the latter in the system, while the basic part of the flow follows to be mixed with the incoming fresh supply of initial stock.

The residue from the bottom of the de-isobutanizer 14, for the purpose of eliminating its butane content, is led into de-butanizer 20. The residue from de-propanizer 19 basically consists of isobutane, which passes heat exchanger 9 and is returned to the reactor, by joining with the flow of the fresh supply of butane-butene fraction.

From the top of de-propanizer 19, the propane fraction is drawn, cooled in condenser 13, and sent into butane-accumulator 21, from where part is returned in the form of spray, while the surplus is led out of the system.

The de-butanized alkylate from the bottom of the de-butanizer

20 is fed into column 22 for rectification.

From the top of column 22, the target aviation component -- the light alkylate, or technical isooctane, is drawn, while from the bottom of the column comes the residue, which is a component of automobile gasoline.

The above-described process is characterized by the following indexes:

Olefins in the initial stock, % by weight	35 - 45
Gram-molecular ratio of isoparaffin to the olefins in the reaction zone	5 - 6
<u>Concentration of acid, in %:</u>	
Fresh supply	98
Used-up	86-87
Volumetric ratio of acid to hydrocarbons in the reaction zone	1-1.8
Maximum temperature in reactor, in °C	10
Contact time, in minutes	20-40
Yield of alkylate, in % by weight of the olefins fed into system	190-200
Yield of aviation alkylate, in % of total combined alkylate yield	90-92
Fresh acid consumed, in kilograms per 1 ton of alkylate yield	170-190
<u>Properties of combined alkylate:</u>	
Specific gravity	0.705
Bromine number	0.3-0.5
Octane number, by the motor method	91
Beginning of distillation, °C	40

10% distilled, up to °C	83
50% distilled, up to °C	106
90% distilled, up to °C	131
End of distillation, °C	206

Properties of fraction boiling up to 150°C(aviation alkylate):

Content of nonsaturated hydrocarbons, in %	Traces
Temperature of 50%, distillate, in °C	104
Reid vapor pressure, in mm of mercury column	181
Octane number, by the motor method	92
Octane number of Grade 1 with the addition of 2.8 milli- liters of tetraethyl lead fluid per liter	104-105
Octane number of Grade 3, with the addition of 2.8 milli- liters of tetraethyl lead fluid per liter	142-143

Consumed per 1 ton of combined alkylate:

Steam, in tons	7.9
Water, in cubic meters	182.0
Electric power, in kilowatt-hours	165.0

[Diagram, page 35 of original]

Flowsheet for the process of sulfuric-acid alkylation  
of isobutene with the aid of butenes

1 - Alkali mixer; 2 - alkali settling tank; 3 - pumps; 4 - acid intake; 5 - contactor (reactor); 6 - alkylation-product discharge; 7 - mechanism for the rotation of impeller in the contractor; 8 - sulfuric-acid settling tank; 9 - heat exchanger; 10 - ammonia cooler; 11 - refrigerating-agent (ammonia) intake; 12 - refrigerant discharge; 13 - condenser; 14 - de-isobutanizer; 15 - preheater; 16 - hot water kettle; 17 - collecting tank; 18 - storage tank for the

circulating isobutane; 19 - de-propanizer; 20 - de-butanizer; 21 - butane collecting tank; 22 - rectification column for the alkylate; 23 - cooler; 24 - collecting tank for light alkylate.

#### THE PROCESS OF HYDROFLUORIC-ACID ALKYLATION OF ISOBUTANE WITH THE AID OF BUTENES

This process of alkylation is conducted in the presence of hydrofluoric acid, which is continuously regenerated in separate apparatus of the same installation. (It must be remembered that hydrofluoric acid is highly toxic. Hence, special safety arrangements are to be followed in the operation of such installations).

Dehydrated initial stock (the butane-butene fraction), intermixed with isobutane, enters, the water-cooled contactors 1, where, at a temperature of 25 - 40°C it is intermixed with the above acid.

The mixture of the hydrocarbons and the hydrofluoric acid follows from contactors 1 into the acid settling tank 3, from where the acid is fed into regenerator 22. From settling tank 3, the mixture of hydrocarbons is fed, via collecting tank 5 and preheater 6, into de-propanizer 7. The propane from the de-propanizer follows into collecting tank 10, from where it is returned as a spray-wash for de-propanizer 7, while its surplus follows into a subsidiary column for the steam-distillation of the acid. The acid at the bottom of collecting tank 10 is drawn into settling tank 3. The propane is led out of the installation, and, following neutralization, is directed into the gas main of the plant. The residue from de-propanizer 7 is fed, via neutralizing bauxite-percolation column 12, into de-butanizer 15, from where the butane follows into cooler 9 and collecting tank 16. Part of the butane is returned as spray-wash

for the de-butanizer, while its surplus is directed into a plant storage tank. The residue from de-butanizer is fed into column 18 for the rectification of the alkylate.

The aviation alkylate from the top of column 18 is drawn into collecting tank 19, and then into a storage tank.

The regeneration of the hydrofluoric acid is accomplished in two stages: in the first stage, the acid is freed from the polymers, in the second stage, it is freed from the water. The elimination of the polymers from the acid is accomplished in regenerator column 22, in which the acid is blown through by isobutane vapors, preliminarily heated in hot-water kettle 8, which operates at the temperature of 180°C. The acid, together with the isobutane, is directed into cooler 9, then into collecting tank 21, and returned to settling tank 3.

The residue from regenerator 22, after cooling, is used as fuel for the plant boilers.

A small portion of the acid is fed from the collecting tank 21 to be dehydrated in subsidiary column 20, from the bottom of which the continuously-boiling water-acid mixture (about 40 percent acid) is drawn. This mixture, after being neutralized with calcium oxide, is led out into the sewer system. The acid drawn from the top of dehydration column 20 follows into settling tank 3, via collecting tank 21.

The production indexes of the process are characterized by the following operating data:

Crude Stock to Processing:

Butenes (including 14 cubic meters of propene and 39 cubic meters of pentenes), in m <sup>3</sup> /24 hours	194
Saturated hydrocarbons (including 67 cubic meters of li- quid isobutane) in m <sup>3</sup> /24 hours	296
Total crude stock, in m <sup>3</sup> /24 hours	490

Circulating isobutane of 80% purity, in m <sup>3</sup> /24 hours	1638
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Obtained:

Aviation alkylate, m <sup>3</sup> /24 hours	329
Heavy alkylate, m <sup>3</sup> /24 hours	21
Isopentane, m <sup>3</sup> /24 hours	30
Normal Pentane, m <sup>3</sup> /24 hours	56
Propane, m <sup>3</sup> /24 hours	17
Acid-dissolved hydrocarbons, m <sup>3</sup> /24 hours	3

total	456
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Yield of aviation alkylate in m <sup>3</sup> per 1 m <sup>3</sup> of olefins	1.7
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Consumed isobutane, in m <sup>3</sup> per 1 m <sup>3</sup> of olefins	1.2
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Operating procedure:

Ratio of isobutane to olefins	7.05
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Ratio of acid to hydrocarbons in reaction zone	1.2
--	-----

Pressure, in atmospheres	10.5
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Temperature, in °C	39
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Time of contact, in minutes	15
-----------------------------	----

Composition of acid:

Acid, determinable by titration in percent by weight	89.6
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Hydrocarbons, in % by weight	3.0
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water, in % by weight	1.6
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Consumed:

Steam, in tons/hour	36
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Electric power, in kilowatts	900
Return water, in cu meters/hour	835
Fresh water added, in m <sup>3</sup> /hour	45
Fuel (gas), in m <sup>3</sup> /hour	24
Caustic soda, in tons/month	3.12
Bauxite, in tons/month	33.0
Hydrofluoric acid, in tons/month	19.0
Calcium oxide, in tons/month	0.5

#### PRODUCTS OBTAINED BY HYDROFLUORIC-ACID ALKYLATION

Physical and Chemical properties	De-pentanized aviation alkylate	Heavy alky- late
Specific gravity	0.7071	0.7981
Beginning of distillation, °C	84	213
10% distilled, up to °C	98	220
50 % distilled, up to °C	107	231
90% distilled, up to °C	123	282
End of distillation, °C	188	324
Reid vapor pressure, in millimeters of mercury column	57	-
Octane number	90	75

[Diagram, page 37 of original]

Flowsheet for the process of hydrofluoric acid alkylation  
of isobutane with the aid of butenes

1 - contractors (reactors); 2 - water-cooling lines; 3- settling  
tank for hydrofluoric acid; 4 - pumps; 5 - collecting tank; 6 -  
preheaters; 7 - de-propanizer; 8 - hot-water kettle; 9 - condensers-

coolers; 10 - collecting tank; 11 - column for the steam-distillation of the hydrofluoric acid; 12- bauxite-percolation columns; 13 - de-isobutanizer; 14 - isobutane tank; 15 - de-butanizer; 16 - collecting tank; 17 - butane tank; 18 - rectification column for alkylate; 19 - collecting tank for light alkylate; 20 - dehydration column; 21 - collecting tank for hydrofluoric-acid regenerator.

#### THE PROCESS OF PHOSPHORIC-ACID ALKYLATION OF BENZENE WITH THE AID OF PROPENE

Cumene, or isopropylbenzene, is an excellent component of aviation gasoline, since it has a high octane number (in its pure state, its octane number, as determined by the motor method is 96), a low point of solidification, and produces better pickup with tetraethyl lead than benzene.

The use of cumene in aviation gasoline permitted the utilization for its production of the sources of coal benzene and of the propane-propene fraction, which was previously not utilized in the production of aviation-gasoline components.

Research resulted in the development of a vapor-phase process of alkylation with the use of a solid catalyst-phosphoric acid on diatomaceous earth.

The process runs as follows.

The crude stock (the propane-propene fraction) and benzene in a predetermined ratio are fed into collecting tank 1. From the collecting tank, the above mixture is forced by pump 2, via heat exchanger 3 and evaporator 4, into reactors 5 (the flowsheet diagram conditionally shows only one reactor) equipped with vertical tubes,



which are filled with the phosphoric-acid treated catalyst. The reactor operates at a temperature of 255-260°C and at a pressure of 25 atmospheres. Having passed through the layer of the catalyst, the mixture emerges from reactor 5, follows through heat exchanger 3, and enters the depropanizer 6. In the depropanizer, the propane and the nonreacted propene are separated and are drawn from the top of the apparatus.

De-propanization is conducted at a pressure of 12 atmospheres and a temperature of about 200°C maintained in hot-water kettle 13.

From the bottom of de-propanizer 6, the residue, which is a mixture of benzene, cumene, and the heavier alkylbenzenes, is drawn into column 9 for the separation of benzene. Column 9 operates at straight atmospheric pressure. The overhead product drawn from this column (benzene) is partly returned to the column in the form of spray, while the remaining quantity of benzene follows into a collecting tank for subsequent circulation.

The residue from column 9 is fed into column 11 for the separation of cumene (isopropylbenzene). The operating cycle of this column is such that only a certain part of the diisopropylbenzene is taken from the top of the column together with the target product-cumene.

Thus, from the top of column 11 is drawn off into a plant storage tank.

If the initial stock used is of adequate purity, no special methods of purification are called for, and the cumene obtained may serve as the commercial component for aviation gasoline.

The vapor-phase process of alkylation is proceeding at relatively low temperatures and pressures. The accessories and the equipment may be made of carbon steel.

For the production of cumene, it is recommended that a benzene containing no more than 0.15 percent of thiophene be used, since the thiophene reduces the life period of the catalyst.

The purification of the benzene from its thiophene content may be accomplished at a temperature of 200°C with the aid of a 98 percent sulfuric acid.

Traces of ethylene and butenes in the propene initial stock are considered to be nondeleterious. Ethylene is slower in entering the reaction than propene, and it passes through the reaction zone without sustaining any changes, while the butenes are almost completely alkylated, resulting in products with properties similar to those of cumene.

#### THE PROCESS IS CHARACTERIZED BY THE FOLLOWING INDEXES

Temperature in the reaction zone, °C	260
Pressure ----, in atmospheres (absolute)	25
Molecular ratio of benzene to propene, in %	10
Volumetric velocity (volume of liquid flow for 1 hr per unit volume of catalyst)	3
Degree of propene conversion, in %	85
Yield of cumene, by the reacted propene, in %	200-225
Consumption of catalyst, in % of the target product	0.3
<u>Properties of the end product:</u>	
Specific gravity	0.8633

Reid vapor pressure, in millimeters of mercury column	1.5
Boiling range for the entire product, °C	138-177
Boiling range for 80 % of the product, °C	146-160
Octane number	99
Flash point, °C	52

[Diagram, page 39 of original]

Flowsheet for the process of phosphoric-acid alkylation of  
benzene with the aid of propene

1 - collecting tank; 2 - pumps; 3 - heat exchanger; 4 - evaporator;  
5 - reactors (only one shown in diagram); 6 - de-propanizer; 7 -  
water cooler; 8 - propane - collecting tank; 9 - benzene-separa-  
tion column; 10 - benzene-collecting tank; 11 -- cumene-separation  
column; 12 - cumene-collecting tank; 13 - hot-oil kettle.

THE PROCESS OF THE VAPOR-PHASE ISOMERIZATION OF NORMAL  
BUTANE

This process permits, by utilizing the isomerization of the  
butane, an increase in the sources of isobutane -- a component that  
must be made available for the production of high-octane alkylates.  
The isomerization of butane is conducted in the presence of a cata-  
lyst -- aluminum chloride, and of anhydrous hydrochloric acid as an  
accelerator.

The initial stock (normal butane) is fed by pump 12 into  
mixer 14, where, for purposes of dehydration, it is intermixed with  
an alkaline solution. The concentration of the alkaline solution  
is 40-45° Baume. The mixture of butane and alkali is allowed to

settle in settling tank 1<sup>7</sup>, the alkalis taken away by a pump and refed into mixer 14, while the butane follows through heat exchanger 16, steam preheater 3, and, in the form of vapors, enters the tubes of reactor 1. The reactors are vertical heat exchangers, the tubes of which contain aluminum chloride. In order to control the temperature of the process, a diathermic petroleum product is circulated in the space between the reactor tubes.

To sustain the activity of the aluminum chloride, the hydrogen chloride which is circulating in the system, is admitted into the butane flow, ahead of its entering the reactors.

The butane, in passing through the tubes containing the aluminum chloride, becomes subject to its catalytic effect, as a result of which the transformation of butane into isobutane takes place.

The flow of the products emerging from reactors 1 passes through adsorbers 4, filled with granulated aluminum oxide, where the aluminum chloride is entrapped, while the remaining reaction products are fed consecutively into condenser 5, cooler 6 with propane as the coolant, and, finally, into collecting tank 7 operating at a pressure of 14 atmospheres. The liquid products from the collecting tank are fed by a pump to column 10 for the distillation of the hydrogen chloride and its subsequent return into the flow of the fresh stock, ahead of reactors 1. Column 10, operating at a pressure of 20 atmospheres is equipped with steam kettle 11. The residue from this column is first cooled in heat exchanger 16, then in cooler 15, following which it is partially drawn for the spraying of scrubber 8. The basic mass of the residue enters mixer 14, where it is neutralized by the circulating alkaline solution from settling tank 13.

The neutralized product (mixture of isobutane, butane, and other) is drawn to rectification for the liberation of isobutane and subsequent return to isomerization of the nonreacted butane.

At the emerging from preheater 3 the temperature is so controlled that following the intermixing of the hydrogen chloride with the butane, the temperature of the total flow ahead of the reactor is above the dew point.

The small amounts of gas generated continuously during the vapor-phase isomerization of normal butane, are released from collecting tank 7, via scrubber 8. In scrubber 8, which is sprayed by the chilled product. (the mixture of isobutane, butane, and other), the hydrogen chloride is entrapped. The gas coming out from the scrubber, consists mainly of methane and small quantities of hydrogen.

The operational indexes, taken by three isomerization installations, and the yield of end products from the single-stage isomerization of butane in its liquid phase, are given below:

#### OPERATIONAL INDEXES

Indexes	Installations		
	A	B	C
[1]	[2]	[3]	[4]
Butane charged into reactor, in			
tons/24 hours	335	360	165
Isobutane content in reaction product, % by volume	47.6	42.5	42.2
Temperature at entering reactor, °C	123	117	101

[1]	[2]	[3]	[4]
Pressure in reactor, in atmos- pheres	18.4	11.3	11.4
Hydrogen chloride content in crude stock, ahead of reactor, molecular %	16.8	13.3	11.0
Catalyst consumed, kg/24 hours	1450	998	500
Aluminum oxide (for adsorber) consumed, kg/24 hours	353	500	308
Hydrogen chloride consumed, kg/24 hrs	-	41.7	41.3
Isobutane obtained, in liters per kilogram of catalyst used	200	250	225
The efficiency factor of the in- stallation (in %)	96.7	91.9	91.7
Volumetric velocity through cata- lyst layer (in reactor), or vol- ume of liquid-stock flow per hour over volume of catalyst used	1.01	0.79	0.84
Volumetric velocity in adsorber, or volume of liquid-stock flow per hour over volume of adsorb- ent used	1.50	2.30	1.83
Butane to isomerization, % by weight		100%	
<u>Reaction products (flow from reactor), % by weight:</u>			
Dry gas		0.7	
isobutane		50.0	
normal butane		48.6	
C5 and heavier		0.7	

[Diagram, page 41 of original]

Flowsheet for the process of the vapor-phase isomerization  
of normal butane

1 - reactors; 2 - coolers for circulating diathermic medium; 3 - steam preheater; 4 - adsorbers; 5 - condenser; 6 - cooler (with propane coolant); 7 - collecting tank; 8 - scrubber; 9 - cooler (with propane as the coolant); 10 - column for hydrogen chloride distillation; 11 - steam kettle; 12 - pumps; 13 - alkali settling tank; 14 - mixer; 15 - cooler; 16 - heat exchanger; 17 - alkali settling tank.

PROCESS OF ISOMERIZATION OF NORMAL PENTANE INTO ISO-  
PENTANE

Isopentane has a high octane number (90, by the ASTM motor method) and a relatively low vapor pressure, as a result of which it may serve as an excellent component of aviation gasoline.

The crude stock (anhydrous normal pentane, free from olefins, and with a benzene content of 0.2%, by volume) is fed by pump 1 into one of the desiccators for dehydration. The desiccators work intermittently: While one is at work, there is regeneration of the desiccant (activated aluminum oxide) in the second one. Upon emerging from the desiccator, the normal pentane is preheated in steam preheater 3, after which it follows into column 4, which is sprayed by the poisoned catalyst.

In the steam preheater, the pentane is heated to a temperature, which is by 15°C lower than the temperature required by the reaction. The further heating is effected through the exothermic heat of the reaction.

Ahead of its entry into the mixer-equipped reactor 5, the following is added to the pentane: hydrogen, hydrogen chloride, and a catalytic mixture (aluminum chloride deposited on granulated bauxite, and antimony trichloride). The pressure in the reactor is sustained at 6-21 atmospheres, and the temperature - at 90 - 95°C. Under these conditions, the reaction mass is in a liquid state.

Under normal operating conditions, a small amount of a complex substance, composed of hydrocarbons and aluminum chloride, is formed. This complex is practically nonsoluble in pentane and is catalytically inert.

In order that the poisoned catalyst does not accumulate in reactor 5, part of it is continuously drawn from the reactor and directed into column 4, from where it is periodically removed. From the reactor, the mixture of the hydrocarbons, antimony trichloride, aluminum chloride, hydrogen chloride, and hydrogen enters column 7 for the separation of the catalyst. All the above components, with the exception of the basic mass of the catalyst, pass out through the top of column 7, while the separated catalyst is pumped into reactor 5, and, partially, by a special pump into saturators 6, where it is enriched with fresh aluminum chloride. Following this, it, together with the basic catalyst flow, is led into reactor 5.

The installation is usually equipped with two saturators working intermittently. They are primed with fresh aluminum chlorides, and are simply pieces of apparatus equipped with steam jackets and functioning at a certain pressure.

The first runnings of column 7 are condensated in cooler 9 and collected in tank 10.



Part of the condensate is returned to serve as a spray for column 7, while the remaining condensate is directed to column 12 for the steam-distillation of the hydrogen chloride. From the collecting tank 10, the basic part of the gases follows into traps 14, from where they are returned, with the aid of compressor 15, into reactor 5.

The part of the gases remaining in collecting tank 10 is passed through absorber 11, which is sprayed by the cold isomerized product (isopentane), the latter absorbing from the gases the remaining hydrogen chloride.

The hydrogen, together with the light gases (methane, propane), is led out through the top of absorber 11, in order to avoid its accumulation, which would produce an accelerated pressure through the system. The hydrogen chloride, together with a small quantity of hydrocarbons, is drawn out as the first runnings from column 12, and returned to reactor 5.

The residue from column 12, which is the isomerization product (isopentane) is sent via cooler 9 into alkali settling tank 13, where it is washed by the alkaline solution, following which it is drawn into a plant storage tank.

Below, the procedure and the indexes of the above-described process are depicted.

Operating conditions in reactor:

Temperature, °C	93
Pressure, in atmospheres	17.6(liquid phase)
Volumetric velocity (the volume of liquid-stock flow per hour over the volume of the impregnated bauxite)	1.0
Concentration of the HCl, in % by weight of crude stock	1.0

Same as above, in % molecular 2.0

Yield of isopentane, in % 60

Catalyst consumption:

$\text{AlCl}_3$  per 1000 liter of iso -  $\text{C}_5$ , in kg 1

or impregnated bauxite per 150 liters of iso-

$\text{C}_5$ , in kilograms 1

[Diagram, on page 43 of original]

Flowsheet for the process of isomerization of pentane into  
isopentane

1 - pumps; 2 - desiccators; 3 - steam preheater; 4 - column with  
poisoned catalyst; 5 - reactor equipped with mixer; 6 - saturators;  
7 - column for the separation of catalyst; 8 - boiling kettles; 9 -  
coolers; 10 - collecting tank; 11 - absorber; 12 - column for the  
steam-distillation of hydrogen chloride; 13 - alkali settling tank;  
14 - gas traps; 15 - hydrogen-chloride compressor; 16 - fresh-  
catalyst storage tank.

THE PROCESS OF ISOMERIZATION OF NARROW GASOLINE FRACTIONS

For the production of isobutane, with a view of increasing its  
sources, the method for the isomerization of normal butane was devised.  
In order to obtain the alkylate, the isobutane, resulting from the  
above isomerization, is directed to alkylation with the aid of bu-  
tenes.

Subsequently, a method was devised for the isomerization of  
the narrow gasoline fractions - the mixture of normal pentane with  
normal hexane.

The isomerization resulted in a mixture of isopentane and neo-hexane, the octane number of which was considerably higher. It is proper to remember the octane number (without the addition of tetra-ethyl lead) of normal pentane is 62, while it is 10 for isopentane, the octane number of normal hexane is 26, while it is 94 for neo-hexane.

The above isomerized high-octane mixtures were being added directly to aviation gasolines.

In the isomerization of the narrow gasoline fractions, the catalyst used is aluminum chloride, which is accelerated by anhydrous hydrogen chloride.

The narrow gasoline fractions, which are sent to isomerization, are usually present in straight-distillation gasolines, their boiling points being close to the boiling point of normal hexane.

Methylcyclopentane (octane number 80) and cyclohexane (octane number 77) are not only excellent components of aviation gasoline, but may also serve as very effective cracking-process decelerators during the isomerization reactions. However, they must not be present in quantities beyond certain limits, since in those cases they may considerably retard the isomerization of the paraffin-base hydrocarbons. This must be taken into account in the fractionation of initial stock to be assigned to isomerization, so that the content of methylcyclopentane and cyclohexane in the above stock is kept within the proper range.

#### A - ISOMERIZATION

The following is indicated in the complex isomerization diagram of the narrow fractions: rectification of the initial stock,

isomerization, the preparation of the catalyst (aluminum chloride solution), and the rectification of the isomerization products.

The initial stock (gasoline fractions) is fed by pump 1, via steam preheater 2, into de-butanizer 4, from the top of which are drawn the butane and the lighter hydrocarbons, which are directed, via water condenser-cooler 5, into collecting tank 6. From collecting tank 6, part of the product is fed in the form of spray for the de-butanizer, while the remaining part is pumped into a storage tank for subsequent utilization.

The residue from debutanizer 4 (the pentanes and the heavier hydrocarbons) are transferred to de-pentanizer 8. The overhead product of the de-pentanizer is pentane, which may be either utilized as automotive fuel, or sent to isomerization.

The residue from de-pentanizer 8 (heptane and the heavier components) is utilized as automotive fuel.

The heptane (side) fraction, emerging along line 9 from de-pentanizer 8, together with the circulating product coming from the top of heptane column 32, is drawn into accumulating tank 10. From tank 10, the mixture being isomerized is pumped through desiccator 11, where its moisture content is removed, then through sand filter 12 for the precipitation of the entrapped particles of calcium chloride, and is finally fed to the top of ring-packed absorber 13.

A gas containing very little hydrogen chloride enters the bottom of the absorber. This gas, after an alkaline wash for the removal of its hydrogen chloride content, is led out from the top, and may be utilized as fuel. Absorber 13 operates at a sustained pressure of 24 atmospheres and at a temperature of about 30°C.

The mixture of the pentanes, hexanes, and the hydrogen chloride from the bottom of the absorber is fed into collecting tank 10, from where it continues, via heat exchanger 14, into steam preheater 2, where it is heated up to 120-150°C, and then directed into the bottom of main reactor 16. Ahead of its entering the above reactor, for the purpose of retarding the nondesirable secondary reactions of cracking, which are setting in during the isomerization process, a flow of hydrogen is introduced into the isomerization stock. From the other end, an aluminum-chloride solution is continuously fed into the reactor by pump 30. The isomerization stock passes through the layer of the liquid catalyst in the reactor, following which it is directed into heated settling tank 17, where the entrapped catalyst is precipitated and discharged into tank 15.

The isomerization products from settling tank 17 are led into immersion cooler 18, chilled settling tank 19, and then into the top of column 20 for the steam-distillation of the hydrogen chloride. From the chilled settling tank 19, a gas, composed primarily of methane, ethane, and some hydrogen chloride is blown off. Column 20 operates at a sustained pressure of 24 atmospheres, and at a temperature of 54°C on top and 180°C on the bottom.

The residue from column 20, after cooling, passes through mixer 21, where it is mixed with an alkali, then precipitated in settling tank 22, follows into second mixer 21, where it is intermixed with water, and, via water-settling tank 23, enters de-butanizer 35.

De-butanizer 35 operates at a sustained pressure of 8 atmospheres and at a temperature of 52°C at the top and 130°C at the bottom.

The overhead product of de-butaniser 35 is a mixture of butanes and the lighter hydrocarbons.

The residue from de-butaniser 35 (pentanes and the heavier hydrocarbons) is directed into hexane column 25.

The column operates at a sustained pressure of 5.3 atmospheres and at a temperature of 128°C at the bottom and 103°C at the top. The overhead (end) product of the hexane column, which is composed of isopentane and neohexane, is led into a plant storage tank.

The residue from column 25 (the hexanes and the heavier hydrocarbons) is directed to the heptane column 32. The overhead product of column 32 is returned to same in the form of spray, while the surplus is sent into collecting tank 10. Part of this overhead product is led into mixer 29 for the preparation of the aluminum chloride solution (catalyst).

The residue from column 32 is pumped along line 33 into a plant storage tank.

The catalyst used in the isomerization of gasoline fractions is a liquid complex which is formed as a result of the reaction of aluminum chloride with hydrocarbons. The liquid complex -- a solution of aluminum chloride, has a specific gravity of 1.3-1.4, a viscosity of  $\eta_{50} = 9$ ,  $\eta_{100} = 2.5$ . Into the composition of this complex enters 35 percent, by weight, hydrocarbons and 65 percent aluminum chloride.

The preparation of the solution is accomplished as follows:

Granulated aluminum chloride is drawn from bin 27, via rotary proportioning feeder 28, into mixer-bin 29, into which the hexane

fraction is also brought in. The circulation of the hydrocarbon and the aluminum chloride is maintained by a high-capacity (about 100 tons/hour) centrifugal pump. The level of the liquid in the mixer-bin is kept constant.

Following multiple recirculation, there is the formation of a complex compound of aluminum chloride and hydrocarbons. From bin 29, the catalyst solution is forced by pump 30 into reactor 16.

[Diagram, page 45 of original]

Flowsheet for the process of isomerization of the narrow  
gasoline fractions

A- The complex diagram for the rectification of the initial stock, the isomerization, the preparation of the catalyst, and the rectification of the isomerization products.

1 - pumps; 2 - steam preheater; 3 - steam kettle; 4 - de-butanizer; 5 - water coolers; 6 - butan-collecting tank; 7 - pentane-collecting tank; 8 - de-pentanizer; 9 - lead-out line for the side (hexane) fraction; 10 - vertical collecting tank for the isomerization stock; 11 -- calcium-chloride desiccator; 12 - sand filter; 13 - hydrogen-chloride absorber; 14 - heat exchanger; 15 - poisoned-catalyst tank; 16 - main reactor; 17 - heated settling tank; 18 - immersion cooler; 19 - chilled settling tank; 20 - hydrogen-chloride steam-distillation column; 21 - mixer; 22 - alkaline settling tank; 23 - water settling tank; 24 - high-pressure water vapor discharge; 25 - hexane column; 26 - collecting tank for isoproducts; 27 - aluminum-chloride bin; 28 - rotary proportioner; 29 - aluminum-chloride mixer-bine; 30 - aluminum-chloride solution pump; 31 - collecting tank; 32 - heptane column; 33 - pumping-off line for naphthene hydrocarbons; 34 - discharge

line for butane and light hydrocarbons to plant storage tank; 35 - de-butanizer for the isoproducts.

#### (B) THE OBTAINING OF ANHYDROUS HYDROGEN CHLORIDE

There are several methods for the obtaining of anhydrous hydrogen chloride.

One of these methods is shown in the diagram.

Sulfuric acid from tank 1 is forced by pump 2 into packed column 3 for the blow-off of the free sulfuric anhydride. With the aid of an inert gas, which is forced through the column into the atmosphere carrying with it the sulfuric anhydride.

The sulfuric acid, deprived of its free sulfuric anhydride, is fed by a pump into a hydrogen-chloride generator, which is made up of two units: the top - a glass-coated column 4 with ring packing, and bottom -- a glass-coated mixer 9. Into this generator (column 4), the sulfuric acid is fed. Below the inlet for the sulfuric acid, there is another inlet, into which hydrochloric acid from tank 10 is pumped.

As a result of the close contact between the sulfuric and the hydrochloric acids on the ring surfaces and in mixer 9, the dehydrated hydrogen chloride is carried upward, enters settling tank 5, follows from there into compressor 6, and then into absorber 13. From the bottom of the mixer, the sulfuric acid (diluted by the moisture eliminated from the hydrochloric acid) is led out and forced by pump 8 into capped column 7, through which an inert gas is blown in order to eliminate the traces of hydrogen chloride. The sulfuric acid is then pumped out and fed to the plant acid-con-



centrating installations.

The approximate operating conditions of the installation, also the yield and the quality of the neohexane, which is the end-product when processing the narrow pentane-hexane fraction with circulation, are given below:

Operating conditions:

Temperature in reactor, in °C	120
Pressure in reactor, in atmosphere	60
Volumetric velocity in reactor, volume of initial stock over volume of catalyst, per hr	1.4
Height of catalyst layer, in meters	6
Hydrogen consumed, in m <sup>3</sup> /m <sup>3</sup> of initial stock charged into reactor	32.3
Hydrogen chloride consumed, in % of weight of initial stock charged into reactor	8
Aluminum chloride consumed, in kg/m <sup>3</sup> of ini- tial stock charged into reactor	4.4

THE YIELD AND QUALITY OF THE NEOHEXANE

Indexes	Products		
	Initial Stock	Circulation 1:1	Circulation 2:1
[1]	[2]	[3]	[4]
<u>Yield, in % by volume:</u>			
dry gas	-	3.6	5.3
butanes	-	6.3	8.7
neohexane	-	87.2	85.2
residue	-	7.0	6.2

[1]	[2]	[3]	[4]
Octane number of product without the addition of tetraethyl lead	65	88.0	91.4

[Diagram, page 47 of original]

B - Flowsheet for the process of obtaining anhydrous hydrogen chloride

1 - sulfuric acid tank; 2 - pumps; 3 - column for the blow-off of the free sulfuric anhydride; 4 - hydrogen-chloride generator (column); 5 - settling tank; 6 - compressor for dehydrated hydrogen chloride; 7 - column for the elimination of hydrogen-chloride traces; 8 - pump for the transfer of the low-concentration sulfuric acid; 9 - mixer; 10 - hydrochloric-acid tank.

## THE PURIFICATION OF CLEAR PRODUCTS AND GASES

### THE REMOVAL OF SULFUR MERCAPTAN FROM GASOLINES

This process requires the use of an alkaline solution containing certain naphthenic acids, cresol, and a certain variety of resin acids generated during the coking of coal. The above additives increase the capacity of the alkaline solution for dissolving the mercaptans. Such is the combined solution used for the purification of gasolines, from which the sulfur mercaptan is to be eliminated. The de-vitalized solution is regenerated and returned into the system.

The gasoline for purification is forced by pump 1 into washing column 2 for the preliminary removal of hydrogen sulfide by the alkali. The alkali-processed gasoline is drawn into vertical settling tank 3, on the emergence from which it is joined by the flow of the solution from collecting tank 11. Both flows enter the bottom of the packed extraction column 4.

The gasoline for purification is drawn upward to meet the counterflow of the regenerated reagent solution coming from collecting tank 5. From the top of column 4 they draw the gasoline into a gasoline settling tank 6, then into oxidation column 14, into which compressed air is fed for the oxidation of the sulfurous compounds, then into filter 15, from where the purified gasoline is drawn into a storage tank.

The devitalized solution of the reagent is drawn into settling tank 7, from where, after passing heat exchanger 9, it enters steam-distillation column 13. The live steam, fed into this column

through line 10, blows off the mercaptans. The solution, which is now free from mercaptans, is drawn from column 13, into regenerator 8, from where, after being regenerated, it is fed into collecting tank 5.

Below are some characteristics of the purification of straight-distillation and of cracked gasolines.

Gasoline	Purifying Mercaptan % of mer- Total Sul- solution content captan e- fur content used, in % liminated in % of purified gasoline			
	[2]	[3]	[4]	[5]
Straight-distillation gasoline				
prior to purification	-	0.091	-	0.27
Straight-distillation gaso-				
line after purification	0.7	0.0025	97.2	0.16
Cracked gasoline prior				
to purification	-	0.0142	-	0.034
Cracked gasoline after				
purification	8.0	0.0010	92.5	0.021

[Diagram, on page 49 of original]

Flowsheet for the purification of gasolines from sulfur  
mercaptan

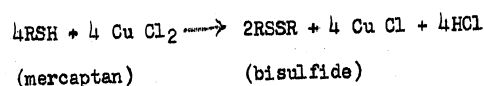
- 1 - pumps; 2 - washing column for the removal of hydrogen sulfide;  
3 - vertical settling tank; 4 - extraction column; 5 - collecting tank

for revitalized reagent; 6 - gasoline settling tank; 7 - settling tank for devitalized reagent; 8 - regenerator; 9 - heat exchanger; 10 - water-vapor intake line; 11 - collecting tank; 12 - water cooler; 13 - steam-distillation column; 14 - oxidation column; 15 - filter; 16 - mixers.

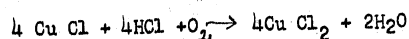
#### THE REMOVAL OF MERCAPTANS FROM GASOLINES WITH COPPER CHLORIDE

The process is developed in two variants: (1) with the use of a copper chloride solution and [2] with the use of a solid reagent, i.e. an absorbent substance impregnated with a copper chloride solution.

The following reactions take place during the purification of gasolines; the mercaptans in the gasoline, reacting with the copper chloride, are converted into Bisulfides, as follows:



The formed copper sesquichloride, air (oxygen)-treated, in the presence of HCl, is reconverted into copper chloride, as follows:



Summarily, the reaction may be represented as follows:



By this method of purification, the conversion of the active (corroding) sulfurous compound - mercaptan - into a nonactive

sulfurous compound-bisulfide, is attained.

To obtain good results, the gasoline to be purified must be subjected to an intimate contact with the copper chloride, while the formed copper sesquichloride and the hydrogen chloride must be thoroughly intermixed with the air that is being fed into the reaction.

In this particular method of purification it is important that the initial gasoline contains neither hydrogen sulfide nor free sulfur.

It must be remembered that the liberated hydrogen chloride in aqueous solution is an active corrosion agent. Consequently, the protection of the apparatus from hydrochloric corrosion is to be provided for.

Theoretically speaking, no reagents are being consumed. For the continuity of the process, the only thing required is the air for the regeneration of the copper chloride.

Practically, the consumption of the reagent is reduced to mechanical losses (carry-offs).

In the first-alternate process, the gasoline to be purified is intermixed with the circulating alkali, and fed into settling tank 2. The preliminary alkaline treatment removes the hydrogen sulfide.

The precipitated gasoline is fed to the suction of the next pump, to which a solution of copper chloride is fed simultaneously from tank 6.

Following the thorough intermixing of the gasoline and the

copper chloride solution, the resulting mixture is drawn into separator 4. From here, the gasoline is piped to a pump, where, after becoming intermixed with the circulating alkali, it is drawn into settling tank 2 for the removal of the copper chloride traces. The purified gasoline is piped from the top of settling tank 2 into commercial tanks.

The solution of copper sesquichloride and hydrogen chloride precipitated in separator 4 is fed continuously into aerator 5. Inside the aerator, there is a series of perforated pipes, the air being fed through the perforations for the purpose of revitalizing the reagent.

The revitalized reagent (copper chloride) is piped into tank 6.

In the second-alternate process, the alkalized gasoline from settling tank 2 is piped into air mixer 8, into which there is a simultaneous feeding of air purified in filter 7.

The gasoline-air mixture follows (in a downward direction) into purification column 9, which is filled with a solid carrier impregnated with copper chloride. The conversion of sulfur mercaptan into bisulfide and the reactivation of the copper chloride take place simultaneously in purification column 9.

The purification installations are equipped with automatic control and measuring instruments.

Data on the purification of straight-distillation gasoline, in comparison with the data on the purification of the same gasoline with a doctor solution, and the consumption indexes, are depicted in the tabulations below.

## RESULTS OF THE PURIFICATION OF STRAIGHT-DISTILLATION

## GASOLINE

Indexes	Initial gasoline	Gasoline purified by copper chloride	Gasoline puri- fied by a doc- tor solution
[1]	[2]	[3]	[4]
Specific gravity	0.7393	0.7393	0.7401
Beginning of distillation, °C	36	37	36
10% distilled up to °C	69	66	71
50% distilled, up to °C	129	127	129
90% distilled, up to °C	200	199	201
End of distillation, °C	227	224	221
Distillate, in %	97.5	97.3	97.1
Residue, in %	0.8	1.0	0.9
Losses, in %	1.7	1.7	2.0
Total sulfur content, in %	0.100	0.101	0.117
Gum in copper cup, milligrams per 100 cm <sup>3</sup>	9	4.8	50.4
Octane number after the ad- dition of 1.5 milliliters of tetraethyl lead per kg	59.9	59.9	58.2
<u>Consumed per 1 ton of gasoline:</u>			
Copper sulfate, in kg			0.00877
Sodium chloride, in kg			0.00879
Sodium sulfide solution, in kg			0.00077
Caustic soda for preliminary alkaline wash, in kg			0.03035



[Diagram, page 51 of original]

Flowsheet for the purification of gasolines by the removal  
of mercaptans with copper chloride

1 - pumps; 2 - settling tanks; 3 - flow controlling device; 4 - separator; 5 - aerator; 6 - copper-chloride tank; 7 - filter; 8 - air mixer; 9 - purification column; 10 - pressure regulator.

THE ELIMINATION OF MERCAPTANS FROM GASOLINES IN THE PRESENCE OF TANNIN

The process described below permits the complete elimination of the sulfur mercaptan from the gasoline.

Caustic soda, which is a good absorbent of hydrogen sulfide, is ineffective for the elimination of the mercaptans (particularly the heavy ones). However, the solubility of the mercaptans in the caustic-soda solution is stepped up when an organic solvent is added to the latter, which, in turn, increases the degree of recovery of the mercaptans from the gasolines.

One of the organic-solvent additives used in this process is the potassium salt of isobutyric acid.

The tannin added to the alkaline solution promotes the oxidation of the mercaptans, soluble in the alkali, into bisulfides, which are nonsoluble in the alkali, by way of air blown through the de-vitalized solution. This permits the effective regeneration by way of oxidation in lieu of using steam for blowing through.

The gasoline to be purified is fed by pump 1 into mixer 2, into which an alkaline solution is pumped simultaneously. The mix-

ture is then piped into alkali settling tank 3. Following the preliminary alkalization (the elimination of hydrogen sulfide), the gasoline flows downward into alkali separator 4 equipped with a distilling head. The alkali and traces of moisture are precipitated in separator 4, the gasoline is piped consecutively through mixer 2 into settling tank 5, in which a three-stage counter flow treatment by alkaline solution with tannin additive is effected.

On the way from the first settling tank to the third, the gasoline is progressively purified by the elimination of the mercaptans, while the counter flow alkaline solution with the tannin additive is being devitalized. From the first settling tank, the devitalized solution is drawn to be regenerated.

Upon emerging from the third settling tank, the gasoline is piped into the tannic-solution separator 6, equipped with a distilling head: The purified gasoline is piped into a plant storage tank, while the separated tannic solution is returned to the third stage of the alkaline treatment.

The regeneration of the tannic solution is accomplished as follows:

The devitalized solution is steam-heated in heat exchanger 14 up to a temperature of 30-60°C and allowed to pass through the distilling head of gasoline separator 13. The separated gasoline joins the flow of the gasoline to be purified ahead of the third stage, while the hot alkaline solution is piped into the upper part of regenerator 12. Into this regenerator is fed a counter flow of air, under the effect of which the mercaptans are converted into bisulfides (reaction of oxidation). The bisulfides and the alkaline

solution pass through the distilling head of bisulfide-separator 11, with the bisulfides drawn out from the top, and the alkaline solution of tannin drawn out from the side and piped into washing column 10. The solution enters column 10 at the top and flows downward, while a counterflow of ligroin is fed through the column for washing out the traces of bisulfides. Then, the ligroin, together with the flow of bisulfides proceeding from separator 11, is drawn out.

The alkaline solution of the tannin is piped into tank 9. From there the regenerated alkaline solution which was precipitated is taken in by a pump and forced through filter 8 and cooler 7. It is then repiped into the system for the 3-stage treatment of the gasoline to be purified.

Data on the elimination of mercaptans from gasolines in the above described purification process is depicted in the table below:

#### THE ELIMINATION OF MERCAPTANS FROM GASOLINE

Mercaptans	Initial gasoline		Purified gasoline	
	Content,	%, to the	Content,	%, to the
	% by weight	total mer- captan	% by weight	total mer- captan
[1]	[2]	[3]	[4]	[5]
Methylmercaptan	0.0165	25.4	-	-
Ethylmercaptan	0.0147	22.7	0.0001	5.0
Polymercaptan	0.0131	20.1	0.0003	15.0
Butylmercaptan	0.0068	10.4	0.0005	25.0
Amylmercaptan	0.0050	7.7	0.0007	35.0
Hexylmercaptan and the heavier mercaptans	0.0089	13.7	0.0004	20.0
Total	0.0650	100.0	0.0020	100.0

[Diagram, page 53 of original]

Flowsheet for the purification of gasolines by the elimination of mercaptans in the presence of tannin

1 - pumps; 2 - mixer; 3 - alkali settling tank; 4 - alkali separator; 5 - tannic-solution settling tank; 6 - tannic-solution separator; 7 - cooler; 8 - filter; 9 - settling reservoir; 10 - washing column; 11 - separator; 12 - regenerator; 13 - gasoline separator; 14 - heat exchanger.

THE ELIMINATION OF MERCAPTANS FROM GASOLINES IN THE PRESENCE OF METHYL ALCOHOL

The considerable increase in the solubility of the higher mercaptans in an alkaline solution in the presence of methyl alcohol dictates the use of this process for the elimination of mercaptans from gasolines.

The gasoline to be purified is piped into column 1 for the washing out of its hydrogen sulfide with alkali, and is then drawn into extraction column 2 for the elimination of the mercaptans. For this purpose, the regenerated alkaline solution is piped by pump 2 from regenerator 6 into column 2. Into the middle part of column 2, methyl alcohol is piped from collecting tank 10.

As a result of the counterflow extraction, the mercaptans are removed from the gasoline by the alcohol-alkaline solution, with the purified gasoline flowing from the top of extraction column 2 into settling tank 3, and from there piped out of the installation. The devitalized alcohol-alkaline solution is piped from the bottom of extraction column 2, via heat exchanger 5, into regenerator 6 for revitalizing.

Into the bottom part of regenerator 6, open steam, preliminarily superheated in steam superheater 11, is introduced. As a result of the interaction between the devitalized solution and the steam, the water vapors and the mercaptans are drawn from the top of the regenerator into condenser-cooler 7 and mercaptan separator 8.

From separator 8, the mercaptans are drawn for subsequent utilization, while the aqueous alcohol solution is piped by a pump into alcohol-rectification column 9 for the extraction of the methyl alcohol.

Open steam is introduced into the bottom of rectification column 9. From the top of the column, the methyl-alcohol vapors are drawn to be condensated in water condenser 7 and follow into collecting tank 10. From collecting tank 10 part of the regenerated methyl alcohol is fed in the form of spray to column 9, while the remaining quantity is again returned into extraction column 2 for the purification of gasoline.

The water from the bottom of column 9 is drawn into the sewer.

Data on the purification of various gasolines, with consumption indexes, will be found in the Table below:

## THE ELIMINATION OF MERCAPTANS FROM GASOLINES

INDEXES

	Straight-distillation gasoline		Mixture of cracked and reformed gasolines				Gasoline from the light cracking of mazut		Cracked Gasoline	
	Initial	Purified	Initial	Purified	Initial	Purified	Initial	Purified	Initial	Purified
	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]	[10]	[11]
[1]										
Gasoline distillation range, °C	38-205	-	54-115	-	57-200	-	27-143	-	38-205	-
Mercaptan content, in %	0.012	0	0.74	0.0095	0.064	0.001	0.27	0.0007	0.058	0.0008
Total sulfur content, in %	-	-	0.11	0.04	0.097	0.034	0.52	0.28	-	-
Octane number of nonethyl gasoline	-	-	79	79	68.5	69.5	71.5	72.5	69.8	70.6
Octane number of gasoline after the addition of 1 milliliter of tetraethyl lead per kg	-	-	-	-	-	-	-	-	75.2	77.0
Octane number of gasoline after the addition of 2 milliliters of tetraethyl lead per kg	76.2	78	81	83.5	78.0	79.5	82.5	80.0	77.5	79.5

[Table continued]

Consumed per 1 ton of gasoline:

High-pressure steam, in tons	0.087
Exhaust, steam	0.064
Water, in Cubic meters	6.0
Electric power, in kilowatt-hours	0.57
Fuel, in kilocalories	20.0
Caustic soda, in kilograms	0.193
Methyl alcohol, in liters	0.166

[Diagram, page 55 of original]

Flowsheet for the process of removing mercaptans from gasolines  
in the presence of methyl alcohol

1 - Alkaline-washing column; 2 - extraction column; 3 - settling tank; 4 - pumps; 5 - heat exchanger; 6 - regenerator; 7 - condenser-cooler; 8 - mercaptan separator; 9 - alcohol column; 10 - methyl-alcohol collecting tank; 11 - steam superheater.

THE PROCESS OF CATALYTIC DESULFURIZATION OF STRAIGHT-DISTILLATION GASOLINES

Granulated bauxite is used as the catalyst in this process. The gasoline to be purified is piped by pump 9, via heat exchanger 2, where it is preheated to a temperature of 280-290°C, into tube heater 3. From tube heater 3, the gasoline vapors having a temperature of 400°C are drawn in a downward direction into catalyst chambers 1 filled with granulated bauxite.

Under normal conditions, the temperature of the gasoline vapors in passing through the catalyst is reduced by 3 - 5°C.

The purified vapors emerge from the catalyst chamber, lose part of their heat content in heat exchanger 2, and with a temperature of 215-220°C are drawn into rectification column 13. The bottom of the rectification column is heated by kettle 11 operating on hot gas oil.

From the top of column 13, the purified gasoline (with a definite ultimate boiling point), which is condensated in heat exchanger 2, is cooled in coolers 7 and is drawn into collecting tank 12. From the bottom of column 13, via cooler 7, the ligroin fraction is drawn.



From collecting tank 12, the purified gasoline is fed by a piston pump, via heat exchanger 2 and 10, into stabilizer 6. From the top of the stabilizer, via collecting tank 8, the nonstable overhead product is piped into a plant storage tank.

From the bottom of the stabilizer, the purified and stable gasoline is drawn.

Under normal operating conditions, one ton of catalyst may pass, without being regenerated, from 300 to 350 tons of gasoline.

Common clay-roasting kilns may be used for the regeneration of the catalyst.

#### RESULTS OF DESULFURIZATION OF STRAIGHT DISTILLATION

##### GASOLINE

Physical and Chemical Properties	Sulfur- containing gasoline	Gasoline after desul- furization	Aviation gasoline fraction after desulfurization
[1]	[2]	[3]	[4]
Specific gravity	0.741	0.7543	0.7397
Color	White	White	White
Doctor Test	Positive	Negative	Negative
Corrosion test	Negative	Negative	Negative
Sulfur content, in %	0.27	0.014	0.008
Reid vapor pressure, in mm of mercury column	470	362	362
Octane number of nonethylated gasoline	59.6	60.9	67.1

[1]	[2]	[3]	[4]
Octane number, with the addition of 1.5 cu cm of tetraethyl lead per liter			
	69.1	81.7	87.3
Beginning of distillation, °C	36	38	40
10% distilled, up to °C	67	67	69
50% Distilled, up to °C	115	107	94
90% distilled, up to °C	178	173	126
End of distillation, °C	196	198	144
Distilled, in %	95.0	97.0	97.0
Residue, in %	1.0	1.2	1.1
Losses, in %	4.0	1.8	1.9

Flowsheet for the process of catalytic desulfurization of  
straight-distillation gasoline

1 - catalyst chambers; 2 - heat exchangers; 3 - tube heater; 4 -  
temperature-control devices; 5 - pressure regulator; 6 - stabilizer;  
7 - water coolers; 8 - collecting tank; 9 - pumps; 10 - heat ex-  
changer operating on hot gas oil; 11 - kettle operating on hot gas  
oil; 12 - collecting tank; 13 - rectification column.

THE PROCESS OF OBTAINING DIESEL FUEL OIL WITH THE USE OF  
LIQUID SULFUR DIOXIDE

Liquid sulfur dioxide, being a solvent, removes from the  
Diesel fuel oil the aromatic and nonsaturated hydrocarbons which  
results in a higher cetane number and also removes compounds con-  
taining sulfur, nitrogen, and oxygen.

It is necessary that the Diesel fuel oil be completely free of  
water, which is accomplished by blowing air through it.

The crude stock is forced by pump 1 through sand filter 2 for the removal of moisture traces and through vacuum deaerator 3 for the removal of traces of air. From the bottom of the deaerator, the crude stock is caught by a pump and piped, via water cooler 4, into calcium-chloride desiccators 5 for the final removal of moisture.

Upon emerging from second desiccator 5, the crude stock passes through heat exchanger 6, tubular cooler 17, and into the bottom of purification column 9. The space between the pipes in cooler 17 contains the liquid sulfur dioxide, the cooling of which is due to evaporation and suction by compressor 14.

The liquid sulfur dioxide is piped from the collecting tank of distributor 12 into cooler 17, from where the cold sulfur dioxide is piped by a pump into the top of purification column 9 equipped with Raschig-ring packing.

From the top of the purification column 9, the refined product is piped into collecting tank 8, from where it is fed, via heat exchangers 6 and 34, into high-pressure evaporator 18 for the regeneration of the sulfur dioxide.

From the bottom of purification column 9, the flow of the extract follows into collecting tank 10, from where it is piped by a pump, via heat exchangers 6, 21, 32 and preheater 31, into high-pressure evaporator 22.

It should be noted that in the production of 50-cetane Diesel fuel oil, the extract solution contains 84%, by weight, of sulfur dioxide, while the refined-production solution contains only 15 percent.

The vapors of the separated sulfur dioxide follow from the

top of evaporator 18 into water condensers 11. The residue from evaporator 18 is drawn into low-pressure evaporator 19. From the top of this evaporator, the sulfur-dioxide vapors enter the intake of compressor 14, are compressed, and are drawn into condensers 11.

The residue from evaporator 19 is drawn into vacuum evaporator 20. The vapors from this evaporator are sucked in by vacuum pump 13 and fed to the intake of compressor 14. The refined product (the Diesel fuel oil) is pumped from the bottom of vacuum evaporator 20.

The sulfur dioxide vapors are drawn from the top of the high-pressure extract evaporator 22, via heat exchanger 30, into a high-pressure collecting and distributing tank 29.

The residue from evaporator 22 is drawn into a medium-pressure evaporator 23, from the top of which the sulfur dioxide vapors are drawn partially into condensers 11, and partially into dewaterer 26.

The residue from evaporator 23 is drawn into a low-pressure evaporator 24, operating at a pressure of only 2 atmospheres.

From the top of evaporator 24, the sulfur dioxide vapors enter into the intake of compressor 14, while the residue is drawn into a vacuum evaporator 25.

From the top of the vacuum evaporator 25, the sulfur dioxide vapors, are drawn out by vacuum pump 13. From the bottom of the vacuum evaporator, the residue (extract) is pumped into a plant storage tank.

The sulfur dioxide vapors are drawn from evaporator 23 into

desiccator 26, which is operating at a pressure of 6.4 atmospheres.

From the top of desiccator 26, the sulfur dioxide vapors are drawn into condensers 11, while water is continuously piped out from the bottom.

#### OPERATING PROCEDURE OF THE INSTALLATION:

Ratio of SO <sub>2</sub> to the product, in %	135
<u>Temperatures, in °C:</u>	
In purification column	(-6)
In the crude-stock cooler (body)	(-3)
In the low-temperature cooler (body)	(-13)
In the main SO <sub>2</sub> collecting tank	33
In the high-pressure SO <sub>2</sub> collecting tank	69
<u>In the extract evaporators:</u>	
In kettle of high-pressure evaporator	118
In kettle of low-pressure evaporator	138
In kettle of vacuum evaporator	151
<u>In the refined-product evaporators:</u>	
In kettle of high-pressure evaporator	118
In kettle of low-pressure evaporator	138
In kettle of vacuum evaporator	149
<u>Pressure:</u>	
In crude-stock cooler, in atmospheres (absolute)	1.4
In low-temperature cooler, in atmospheres (absolute)	0.9
In top of extraction, column, in atmospheres (absolute)	1.4
In high-pressure extract evaporator, in atm (excess)	1.4
In feed-line to high-pressure condensers, in atm (excess)	-5.6
In vacuum evaporator, in mm of mercury column (absolute)	60

# **YIELDS AND CHARACTERISTICS OF CRUDE STOCK, AND PRODUCTS IN THE REFINING OF DIESEL FUEL OIL**

Indexes	Crude Stock	Extract	Refined Product
[1]	[2]	[3]	[4]
Yield, volumetric %	-	25.0	75.0
Cetane number	40	-	50
Flash point, °C	82	82	82
Viscosity, $\text{cP}_{38}$	1.12	1.12	1.12
Solidification point, °C	-34	-	26
Sulfur content, %	0.34	1.25	0.02
Aromatic and nonsaturated hydrocar- bons, %	24	78	3
Specific gravity	0.8612	0.9297	0.8338
Beginning of distillation, °C	199	202	201
10% distilled, up to °C	230	229	230
50% distilled, up to °C	254	255	256
90% distilled, up to °C	297	296	297
End of distillation, °C	324	331	323

[Diagram, page 59 of original]

Flowsheet for the process of obtaining Diesel fuel oil

with the use of sulfur dioxide

1 - pumps; 2 - sand filter; 3 - deaerator; 4 - water cooler;  
5 - desiccators; 6 - heat exchangers; 7 - separator; 8 - refined-  
product collector; 9 - purification column; 10 - extract-collecting  
tank; 11 -- water condensers for sulfur dioxide; 12- collecting

and distributing tank for sulfur dioxide; 13 - vacuum pump; 14 - compressor; 15 - low-temperature cooler working at atmospheric pressure; 16 - piston pump; 17 - low-temperature cooler working at a pressure of 1.4 atmospheres; 18 - high-pressure refined-product evaporator; 19 - low-pressure evaporator; 20 - vacuum evaporator; 21 - steam-distillation heat exchanger; 22 - high-pressure extract evaporator; 23 - medium-pressure extract evaporator; 24 - low-pressure evaporator; 25 - vacuum evaporator; 26 - desiccator; 27 - steam kettles; 28 - preheater; 29 - high-pressure collector-distributor for sulfur dioxide; 30 - steam distillation heat exchanger; 33 - water cooler; 34 - heat exchanger for refined product; 35 - cooler.

#### PURIFICATION OF GASES BY THE REMOVAL OF HYDROGEN SULFIDE WITH AMINOSOLUTIONS

The process is used for the precipitation of hydrogen sulfide and carbon dioxide from gases and liquid products by washing with aminosolutions (organic alkali), which carry off the acid gases. The absorbed acid gases are precipitated from the solution by heating, and the revitalized aminosolution is returned into the cycle. The revitalizing of the solution occurs due to the fact that the amino-salts of milk acids are readily decomposed under the effect of accelerated temperatures, and the amines may be separated from the acid gases absorbed at lower temperatures.

The process is used for the:

- (1) Purification of natural gas,
- (2) Purification of gases in Petroleum Refining,
- (3) Purification of liquid hydrocarbons,

- (4) Purification of hydrogen,
- (5) Purification of carbon dioxide,
- (6) Precipitation of carbon dioxide,
- (7) Precipitation of hydrogen sulfide.

The gas to be purified enters collecting tank 1, and then absorber 3. The absorber is sprayed with a revitalized aminosolution, with the amines absorbing the hydrogen sulfide from the gas.

The purified gas is drawn from the top of the absorber into collector 2, from where it is drawn for utilization. The devitalized aminosolution from the bottom of absorber 3 is directed, via heat exchanger 6, into the top of desorber 8. Heat is imparted to the solution with the aid of steam kettle 9.

As a result of this heat treatment, the revitalized alkaline aminosolution follows from the bottom of the desorber, via heat exchanger 6, to the intake of pump 5. This pump feeds the solution into absorber 3 for the absorption of the hydrogen sulfide.

Indexes for the purification of various gases with aminosolutions are depicted in the Table below:

INDEXES FOR THE PURIFICATION OF GASES BY THE REMOVAL OF  
HYDROGEN SULFIDE

Indexes	Installation	Installation	Natural	Purification
	flue gases	flue gases, gas		and dehydra-
	with high con-	with low		tion of na-
	tent of hy-	content of hy-		tural gas
	drogen sul-	drogen sulfide		
	fide	with close to		
		dew point tem-		
		perature		
[1]	[2]	[3]	[4]	[5]



[1]	[2]	[3]	[4]	[5]
Amount of gas purified, in thousands of m <sup>3</sup> /24 hours	130	150	227	285
Temperature of gas, °C	32	68	23	16
Pressure in absorber, atmospheres	7	20	28	28
H <sub>2</sub> S content (including mercaptans of initial gas, grams/ 100 m <sup>3</sup>	5,010	690	373	300
H <sub>2</sub> S content (including mercaptans) in the purified gas, in grams/100cm <sup>3</sup>	183	21	-	-
H <sub>2</sub> S content in the purified gas, in grams/100 cm <sup>3</sup>	25	-	0	0
Amount of solution fed, in liters/min	284	81	85	51
Amines in solution, % by weight	9.5	13.5 <sup>(1)</sup>	5.6 <sup>(2)</sup>	11.1 <sup>(2)</sup>
H <sub>2</sub> S content in revitalized solution in mg/liter	599	274	582	137
H <sub>2</sub> S content in saturated solution, in mg/liter	15,578	10,032	8,423	14,509
Steam consumed for revitalizing solution, kg/hour	1,310	278	-	360
Steam consumed, kg/kg of H <sub>2</sub> S removed	4.8	6.6	-	9.2
Steam pressure in kettle, in atmospheres	0.5	0.7	0.5	2.6
Pressure in regenerator, in atmospheres	0.14	0.4	0.07	0.07

[1]	[2]	[3]	[4]	[5]
Temperature of solution entering absorber, °C	31	60	34	26
Temperature of solution leaving absorber, °C	32	65	28	15
Temperature of solution entering regenerator, °C	91	97	80	90
Temperature of solution leaving regenerator, °C	106	111	102	129
Amine replenishment, kg/24 hrs <sup>(3)</sup>	3.2	6.9	-	0.5
Lowering of dew point, °C	-	-	-	19

- (1) Diethanolamine
- (2) Monoethanolamine
- (3) Total loss, including evaporation, chemical, and mechanical losses.

[Diagram, page 61 of original]

Flowsheet for the removal of hydrogen sulfide from gases  
with aminosolution

1- gas-collecting tank; 2 - collecting tank for purified gas; 3 - absorber; 4 - water cooler; 5 - pump; 6 - heat exchanger; 7 - gas separator; 8 - desorber; 9 - steam kettle.

THE PROCESS OF PHENOLATE PURIFICATION OF GAS

This process is used for the removal of hydrogen sulfide from hydrocarbon gases. The gas purification is usually effected in distilling-head columns.

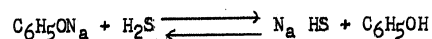
The process is continuous, with simultaneous regeneration

of the devitalized solution and precipitation of hydrogen sulfide for subsequent utilization.

The process is based on the fact that hydrogen sulfide reacts readily with caustic soda at normal temperature, while the resulting compounds do not disintegrate at accelerated temperatures.

To attain the regeneration of the devitalized solution, it is necessary to add to the latter an acid component. The acidity of this additive at normal temperature is to be lower than the acidity of the hydrogen sulfide, yet, when heated, it is to increase at the same rate as the acidity of the hydrogen sulfide. Phenol is the substance that specifically fulfills these requirements. Hence, when adding phenol into a caustic-soda solution, resulting in the formation of sodium phenolate, the hydrogen sulfide displaces the phenol at normal temperature.

By bringing the devitalized solution to the point of boiling, the phenol acquires a higher acidity and displaces the hydrogen sulfide in the form of gas). The reaction is practically fully reversible.



At low temperatures, the reaction proceeds from left to right, while at high temperatures (as prevail during the solution-regeneration cycle)-- from right to left.

The diagrammatic description of the two-stage gas-purification process follows.

The gas to be purified enters the bottom of first absorber 11 and passes through the entire height of first absorber 11 and

second absorber 12. The purified gas is drawn from the top of the second absorber.

The partly regenerated solution is piped from the bottom of first desorber 5, and, upon passing through heat exchanger 10, enters the intake of a pump, which forces the somewhat cooled solution, through water cooler 9, into the top of the first (lower) absorber 11.

Simultaneously, the fully regenerated solution from the bottom of second desorber 6 enters under pressure into cooler 9, and then into the intake of a pump, which drives it into the top part of second absorber 12.

The devitalized phenolate solution under the absorber pressure (the pressure maintained in the absorber is generally higher than that in the desorber) enters heat exchanger 10, and, then, separator 8. Upon emerging from separator, the flow of the solution is split up and sent to regeneration into the top of second desorber 6 and into the top of first desorber 5.

The temperature in the top of the first desorber is regulated with the aid of a spray proceeding from water-cooled dephlegmator 4. Also, from this dephlegmator, the concentrated hydrogen-sulfide fumes are led out for subsequent utilization.

Kettle 3 serves for the boiling of the solution.

The approximate material balance of the process and its consumption indexes are depicted below:

Crude gas from cracking                      to purification → 100.0%  
Obtained:

Purified gas	95.0%
Hydrogen sulfide	4.0%
Losses	1.0%

Consumed per ton of purified gas:

NaOH, in kilograms	0.16
Phenol, in kilograms	0.3
Steam, in kilograms	203
Water, in m <sup>3</sup>	5

[Diagram, page 63 of original]

Flowsheet for the process of phenolate purification  
of gas

1- Phenolate-solution tank; 2 - pump; 3 - kettle; 4 - dephlegmator; 5 - first desorber; 6 - second desorber; 7 - connector; 8 - separator; 9 - coolers; 10 - heat exchanger; 11 - first absorber; 12 - second absorber.

## THE PRODUCTION OF OILS

### THE PROCESS OF DE-ASPHALTING OIL WITH PROPANE

When oil is dissolved in liquid propane, there occurs the coagulation of the asphalts and the tars, as a result of which it becomes possible to eliminate these from the solution.

An essential condition for the process is the quantity of the solvent and the proper temperature of the mixture.

The addition of a small amount of propane to tar oil results only in the reduction of the viscosity of the latter without a perceptible precipitation of the asphalts.

When increasing the amount of propane added, upon attaining a definite (critical) volume (in relation to the oil), the oil will split into two phases: a heavy high-viscosity asphalt phase and a fluid propane phase. The asphalt, from which the propane was removed, has a low melting point, due to the presence of oil in it. The oil separated from the propane solution will contain a certain amount of tar substances.

When adding a considerable amount of propane, an asphalt of high viscosity and of a high melting point can be obtained simultaneously, the oil thus obtained will contain a very small amount of asphaltic and tarry substances.

On arriving at a certain ratio between the volumes of the propane and the oil, a maximum propane effect sets in, and any subsequent addition of propane to the oil will practically have no effect upon the tar content of the latter.

The temperature cycle affects perceptibly the amount and the

nature of the asphaltic and tarry substances precipitated. Within the limits of  $-42$  to  $+20^{\circ}\text{C}$ , propane will dissolve paraffins and ceresins to a rather small degree only. At temperatures of  $40-60^{\circ}\text{C}$ , asphaltic substances are very difficultly soluble in propane, while at the same time oil and paraffins are readily soluble in it. In the interval  $40-100^{\circ}\text{C}$ , the solubility of oil in propane diminishes gradually with the increase in temperature, with the high-molecular hydrocarbons the first to be precipitated from the solution. Thus, by selecting the necessary conditions (temperature, pressure, concentration), account also being taken of the various degrees of solubility in propane of the oil fraction to be extracted, the latter may be divided into two or several parts.

The crude stock is piped by pump 1 into extraction column 4, the upper part of which contains a steam preheater 5. Simultaneously, propane is fed into the middle and bottom parts of this column. Following the counterflow extraction, the solution of the oil in the propane is drawn off from the top of the column and fed, via two vertical steam preheaters 6, into evaporator 8. In order to maintain the required temperature in the evaporator, there is a steam preheater at the bottom.

From the top of evaporator 8, the steam-distilled propane enters condenser-cooler 2, then propane collecting tank 17, from where the propane is returned, with the aid of a centrifugal pump, into extraction column 4.

The residue (asphalt-tar-propane mixture) is piped from the bottom of the extraction column into tube heater 10, and then into evaporator 12 for the steam-distillation of propane. The residue

(extract), freed from the basic mass of propane, is forced by a piston pump from the bottom of steam-distillation column 13, via immersion cooler 18, into a plant storage tank.

The de-asphalted oil with some small content of propane is fed, via preheater 3, into steam-distillation column 9. The propane vapors and the water vapors enter mixing condenser 14, into which water is fed through line 15. The liberated propane is drawn into water separator 16, and then into the intake of propane compressor 19, the compressed propane following into cooler 2. The residue from column 9 (de-asphalted oil) is forced by a pump, via tubular cooler 2, into a plant storage tank.

Examples of deasphalting oil concentrates of typical petroleum varieties are depicted in the Table below: [See table on following page]

The process of deasphalting with propane is also used when preparing weighted crude stock for the catalytic cracking by way of deasphalting straight-distillation maxuts [petroleum residues].

As an example, the data on the deasphalting of two varieties of mazuts is depicted below:

Characteristic of crude stock

Specific gravity	0.9415	0.9516
Viscosity, $^{\circ}\text{S}$ 100	5.0	-
Coke, as per konrdsen, in %	5.92	7.20
Content of salts, mg/liter	302	622
Freezing point, $^{\circ}\text{C}$		32

Operational Cycle:

Propane-crude stock ratio	4.33	5.2
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## DEASPHALTING OIL CONCENTRATES OF TYPICAL PETROLEUM VARIETIES

Crude Stock and Product	Concentrate			Bitumen				
	Yield in %	Specific gravity	$\gamma_{100}$	Coke in %	Specific gravity	Melting point, °C	Pene- trating	Ductility
[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]
<u>Pennsylvania oil concentrate:</u>								
Crude	100	0.908	6.28	3.1	-	-	-	-
deasphalted	98	0.901	4.83	1.6	1.004	51	-	-
<u>Oklahoma heavy oil concentrate:</u>								
Crude	100	0.959	15.66	10.3	-	-	-	-
deasphalted	65	0.914	4.03	1.26	1.054	74	4	0
<u>Emben oil concentrate:</u>								
Crude	100	-	5.41	3.8	-	-	-	-
deasphalted	85	-	3.59	1.5	1.0	60	-	-
<u>Baku II oil concentrate:</u>								
Crude	100	-	32.5	20.0	-	-	-	-
deasphalted	40	-	4.95	1.75	1.04	-	-	-
<u>Baku II oil concentrate:</u>								
Crude	100	-	6.1	11.5	-	-	-	-
deasphalted	55	-	1.84	1.0	1.05	70	6	1

Pressure, in atmospheres	36.9	35.5
Temperature, °C	63	65

Yield of Products:

Deasphalted gas oil, % by volume	92.44	69.74
Petroleum asphalt, % by volume	7.56	30.26

Characteristic of products:

## Gas oil:

Specific gravity	0.9279	0.9260
Viscosity, $\frac{\text{g}}{\text{cm}^2} \text{ at } 100^\circ\text{C}$	3.4	-
Flash point, °C	196	107
Coke, as per konradsen, %	2.87	1.98
Content of salts, mg/liter	6	6
Freezing point, °C	-	43

Petroleum asphalt:

Specific gravity	1.0442	-
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[Diagram, page 65 of original]

## Flowsheet for the process of deasphalting oils with propane

1 - pumps; 2 - water cooler; 3 - steam preheaters; 4 - extraction column; 5 - steam preheater; 6 - vertical steam preheaters; 7 - water vapor feed line; 8 - evaporator for propane-oil mixture; 9 - steam-distillation column for deasphalted oil; 10 - tube heater; 11 - fuel feed line; 12 - evaporator for asphalt-propane mixture; 13 - distillation column for the separation of propane from asphalt; 14 - mixing condenser; 15 - water feed line; 16 - water separator; 17 - propane - collecting tank; 18 - immersion cooler; 19 - propane compressor.

## THE PROCESS OF SELECTIVE REFINING OF OILS WITH THE AID OF VAPOR SOLVENTS

In this process, not one solvent, but two nonmixing solvents are used: (a) propane and (6) a mixture of 30-50 percent phenol with 70-50 percent cresol.

The crude stock (oil concentrate is forced by a pump from reservoir 35 into mixer 24, into which simultaneously enters a refining solution from settling tank 22 and an extract solution from settling tank 26.

Upon emerging from mixer 24, the mixture is guided into cooler 23, and then into settling tank 25.

The crude stock is subject to counterflow extraction, consecutively in extractors - settling tanks 25, 26, 27, 28 and 29.

While the crude stock enters, via mixer 24, the settling tank 25, propane is fed from tank 33 into first settling tank 21, and a mixture of phenol and cresol is fed into the seventh settling tank 29 from tank 30. The upper layer forming in the settling tanks, under the pressure developed by the pump and the injection effect of the mixers, moves in the direction from settling tank 21 toward settling tank 29, while, at the same time, the bottom layer, being forced by pumps from one settling tank to the other, moves in the opposite direction.

From the last settling tank 29, the solution to be refined emerges, as the upper layer, and follows into propane-refining column 8. From the first settling tank 21, the extract solution emerges, as the bottom layer, and follows, via heat exchangers 9, into

propane-extraction column 15.

From the top of column 8, the propane vapors are led out, while from the bottom of same (the steam-distillation section) column -- the mixture of the raffinate with phenol, cresol, and a negligible quantity of the nonsteam distilled propane is led out.

The temperature in the bottom of the column, which is required for the maximum degree of steam-distilling the propane, is maintained by the flow of the hot residue from column 8 that circulates through a section of tube heater 7. The residue from column 8 is passed, via a reduction fitting, into the concentration part of column 6, operating at a pressure of 1.5-1.8 atmospheres (absolute). From the top of this column, the phenol and cresol vapors are led into dehydration column 13.

The residue from the bottom of column 6 is forced by a centrifugal pump through a section of tube furnace 7, and partially fed to steam-distillation column 5 for the final steam-distillation of the phenol and cresol, and partly into the middle section of column 6. The phenol and cresol vapors from column 5 are drawn into the bottom section of column 6. The residue the raffinate is forced by piston pump 4 from column 5, via steam generator 3 and immersion cooler 2, into tank 1.

The extract solution from settling tank 21 passes through two heat exchangers 9 and enters into propane-extraction column 15, which operates at a pressure of 15-18 atmospheres (absolute). The propane vapors are drawn from the top of the column, and together with the propane from column 8 are led into cooler 34, and from there into propane tank 33.

The residue from column 15, via a reduction fitting, is drawn into column 17 for the distillation of the phenol and the cresol. The operating pressure in column 16 is maintained at 1.5-1.8 atmospheres (absolute).

The vapors of phenol, cresol, and water are drawn from the top of column 17 into heat exchanger 9, and then, upon merging with the vapors of phenol, cresol, and water from column 6, are drawn into dehydration column 13. From the top of this column emerges the azeotropic mixture of the vapors of phenol, cresol and water.

In the vertical water condenser 12, the vapors of the azeotropic mixture are condensated and enter separator 11. The vapors of the separated propane are drawn through the top of the separator into trap 31, from where they follow into the intake of compressor 32, which compresses same to 20 atmospheres. The compressed propane, via water cooler 34, is drawn into tank 33.

The solution of water in the phenol and cresol is drawn from separator 11 as a spray for column 13. The bottom layer from separator 10 is forced by a pump into the propane line and, together with the propane, enters cooler 34, and then tank 33.

The residue (extract) from column 17 is pumped through furnace 16, and from there partly to steam-distillation column 18, and partly to column 17.

From the final blowoff of the phenol and cresol from the extract, free superheated steam is introduced into the bottom of column 18 from steam-generator 3. The vapors of phenol, cresol and water are drawn from the top of column 18 into column 17.

The extract from the bottom of column 18 is pumped through steam generator 3, then cooled in an immersion cooler, after which

it enters the tank for the extract.

The quality of the crude stock, the yield and the quality of the raffinate, after deparaffinisation and purification with clay of the various crude-stock residues, are depicted in the Table below :

#### QUALITY OF THE CRUDE STOCK, YIELD AND QUALITY OF PRODUCT

Indexes	P e t r o l e u m   r e s i d u e s				
[1]	[2]	[3]	[4]	[5]	[6]
<u>Crude stock:</u>					
Specific gravity	0.904	0.920	0.935	0.945	0.950
Viscosity, $\vartheta$ 100	2.95	3.04	10.40	6.19	12.0
Viscosity-weight constant	0.827	0.847	0.849	0.864	0.865
<u>Raffinate:</u>					
Yield, in %	83.4	75.5	67.6	58.8	40.0
Specific gravity	0.8789	0.8789	0.8855	0.880	0.900
Viscosity, $\vartheta$ 100	2.25	1.95	3.64	2.75	3.0
Viscosity-weight constant	0.800	0.804	0.800	0.797	0.810
Extract, specific gravity	1.032	1.047	1.039	1.031	-

[Diagram, page 67 of original]

Flowsheet for the process of selective purification of oils with vapor solvents

1- Tank for the refining product of purified oil; 2 - immersion cooler; 3 - steam generator; 4 - pump; 5 - steam distillation column; 6 - column for the separation of phenol and cresol from the raffinate; 7 - two-section tube heater; 8 - propane-refining column; 9 - heat exchanger; 10 - separator for the solution of water, phenol,

and cresol; 11 - separator for the two layers: phenol and cresol; in the water, and water in the phenol and cresol; 12- water condenser; 13- dehydration column; 14 - phenol and cresol cooler; 15 - propane extraction column; 16 - tube heater; 17 - column for the separation of phenol and cresol from extract; 18 - steam-distillation column; 19 - immersion cooler; 20 - water cooler; 21,22- extractors-settling tanks; 23 - mixture cooler; 24 - mixer; 25 - 29 - extractors-settling tanks; 30 - phenol and cresol tank; 31 - trap; 32 - propane compressor; 33 - liquid-propane tank; 34 - propane condenser-cooler; 35 - crude-stock tank 36 - tank for the nonrefinable products; 37 - tank for the extract.

#### THE PROCESS OF REFINING OILS WITH FURFUROLE

Furfurole as a selective solvent is used for the refining of the distillate and residual oils, which were preliminarily deasphalted with propane. Due to its high specific gravity (1.16), furfurole is easily separated from the oil being refined, while its relatively low boiling point (about 160°C) permits its regeneration from the raffinate and from the extract by distillation.

The crude stock, at a temperature of 40-100°C, is forced by pump 1, via heat exchanger 2 and preheater 4, into Raselig-ring packed counterflow-extraction column 5. Furfurole, heated to 90-120°C is fed into the top of this column by a pump from collecting tank 12. A temperature differential of 15-50°C is maintained between the top and the bottom of the column. Due to the difference in the specific weights, the distillate rises to the top of the column, while the furfurole descends to the bottom.

The extract solution, containing a considerable quantity of

furfurol, is continuously drawn from the bottom of the column into collecting tank 7, while the solution to be refined, containing a small amount of furfureole, is drawn out through the top of extraction column 5 into collecting tank 6. From collecting tank 6, the refining solution is forced by a piston pump through heating section 20, and, with a temperature of about 200°C, it enters vacuum refining column 15.

The top section of column 15 is sprayed with furfureole (part of the flow) from collecting tank 12. The furfureole and water vapors are condensated and cooled in cooler 3, and follow into vacuum receiver 8. From this receiver, the water with the furfureole is fed to the top of column 13, from where the water vapors with the negligible amount of furfureole are drawn into cooler 3, condensated and directed into receiver 9. Part of the condensate is returned to column 13, while the remaining part is drawn into column 10 for dewatering. Into the same column, along line 11, is introduced water vapor for the steam-distillation of the furfureole traces. From the bottom of column 10, water is continuously discharged into sewer. The dewatered furfureole, is drawn from column 13, through a side flow, into collecting tank 12, from where it is driven by a centrifugal pump, via heat exchangers 2 and cooler 3, into the top of extraction column 5.

From the bottom of refining column 16, via heat exchanger 2 and cooler 3, the refined oil is continuously driven off by a piston pump.

The extract solution is driven from collecting tank 7, via several heat exchangers 2 and vertical heat exchanger 14, by a centrifugal pump into column 13 for the distillation of the furfureole.



In vertical heat exchanger 14, there occurs the utilization of the latent heat forming in the condensation of vapors, coming from the top of column 17, which is sprayed with furfurole from collecting tank 12.

From the bottom of column 13, via heating section 21, the extract solution is forced by a centrifugal pump into high-pressure extraction column 17.

From the bottom of column 17, the extract, a considerable part of which is drawn into vacuum column 18, is drawn into the intake of a piston pump, which feeds it, via a centrifugal pump, into the bottom of extraction column 5. The vapors from vacuum column 18, together with the vapors from vacuum column 15, enter vacuum receiver 8. From the bottom of steam-distillation column 19, via heat exchanger 2, the extract is continuously pumped out.

The basic furfurole mass is distilled from the extract in column 17, from which, in the form of vapors, it enters vertical heat exchanger 14. In the heat exchanger, the vapors are condensed and, in the form of dewatered furfurole, enter collecting tank 12.

Below- there is data relating to the approximate temperature cycle of an oil-refining installation, and the results of refining a number of oils.

Temperature cycle:

Temperature in top of extraction Column, °C	100
Temperature in bottom of extraction column, °C	75
Temperature in extract layer after cooling, °C	40
Temperature in top of refining column, °C	98

Temperature in bottom of refining column, °C	165
Temperature in top of extraction column, °C	162
Temperature in lower part of top of extraction column, °C	187
Temperature in top of steam-distillation column, °C	98
Temperature of flowing off extract, °C	135
Temperature of heating refining solutions in furnace, °C	200
Temperature of heat extract solution in furnace, °C	230
Temperature of heating crude distillate, °C	80
Temperature of heating furfurole entering extraction column, °C	100
Temperature of regenerated furfurole, °C	155 - 160

## RESULTS OF THE REFINING OF OILS

Indexes	Crude stock	Raffin-ate	Crude stock	Raffin-ate	Crude stock	Raffin-ate	Crude stock	Raffin-ate
[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]
Specific gravity at 15°C	0.941	0.910	0.925	0.893	0.934	0.915	0.912	0.870
Viscosity index	-3.5	52	30	63	25	50	77	108
Petroleum coke as per kondradsen	0.14	0.04	0.20	0.02	0.75	0.28	0.94	0.12
Solidification point °C	-20	-17	-12	-6	-11	-1	+35	+45
Raffinate yield, %	-	61.5	-	69	-	75	-	67.5
Furfurole, % by volume	-	206	-	200	-	150	-	230

[Diagram, page 69 of original]

1 - pumps; 2 - heat exchangers; 3 - coolers, 4 - preheater; 5 - counterflow extraction column; 6 - refining-solution collecting tank; 7 - extract-solution collecting tank; 8 - vacuum receiver; 9 - receiver; 10 - dewatering column; 11 - water vapor feed line; 12 - furfurole collecting tank; 13 - furfurole column; 14 - vertical heat exchanger; 15 - vacuum refining column; 16 - steam-distillation column for raffinate; 17 - high-pressure extract column; 18 - vacuum extract column; 19 - steam-distillation column for extract; 20 - heating section for refining solution; 21 - heating section for extract solution; 22 - furfurole feedline from tank; 23 - vacuum pump.

#### THE PROCESS OF REFINING OILS WITH PHENOL

The use of phenol as a selective solvent in the refining of oil is widespread.

The phenol is used for the purification of the distillates and residual oils following their preliminary deasphalting with propane.

The crude stock is forced by pump 1, via heat exchanger 2 and steam preheater 3, into absorber 4, into which also enters a mixture of water vapors and phenol vapors.

In absorber 4, the crude stock absorbs the phenol and, together with it, is fed into extraction column 5. From tank 18, via steam preheater 3, the dewatered phenol is drawn into the top of column 5.

Into the bottom of this column, from tank 6, enters the phenol

water (up to 10 percent phenol). The refining solution is collected into tank 17, from which, via heat exchanger 2 and tube heater 16 at a temperature of 200°C, it is fed into the top of refining column 8.

Here, most of the phenol is distilled and then drawn, via heat exchanger 2 and cooler 14, into tank 18 for the dewatered phenol.

The raffinate containing some phenol is drawn from the top into the bottom of column 8, operating under a vacuum.

Open steam is introduced into the bottom of the column.

The phenol and water vapors are drawn from the upper part of the bottom of the column.

The residue from column 8 (the refined product) is forced out by a pump and is fed, via heat exchanger 2 and cooler 14, into a storage tank.

The extract solution from the bottom of column 5, at a temperature of 60-80°C is fed, via heat exchanger 2, into the bottom of phenol column 10. In this section of the column, in the form of an azeotropic mixture with phenol, all the water is distilled.

From the bottom of column 10, the mixture of the dewatered phenol and the extract is pumped into the upper section of the same column for the distillation of the basic mass of the dewatered phenol, the vapors of which enter kettle 9, are condensated, pass through heat exchanger 2, are cooled in cooler 14, and enter tank 18. The extract solution circulates through furnace 15, where it is heated to a temperature of 260-270°C. This temperature permits the distilla-

tion of a considerable part of the phenol in the top section.

The extract, containing most of the phenol, is drawn from the lower part of the top section of column 10 into steam-distillation column 13, operating under a vacuum, where the phenol is distilled by free steam.

The extract is forced by a piston pump, via cooler 14, into a tank. The vapors from the bottom section of refining column 8 and from column 13, which contain some phenol, are condensated in water condenser 7, and, in the form of phenol water, flow down into tank 11, from where the damp phenol is pumped into the bottom section of phenol column 10. The phenol containing vapors from the bottom section of phenol column 10 are drawn into cooler-condenser 7, are condensated, and, in the form of phenol water, pumped into the bottom of extraction column 5. The remaining part of the nontrapped phenol and water is drawn into absorber 4, which is sprayed by the crude stock.

The approximate operational cycle of an installation and the result of refining various oils is given below.

Operational cycle of basic apparatus:

Temperature of raw stock when entering extraction column, °C	40
Temperature of phenol when entering extraction column, °C	80
Temperature of Refining solution when emerging from furnace	
°C	240
Temperature of extract solution when entering the top section of phenol column, °C	195
Temperature of extract solution when emerging from furnace °C	270
Temperature in top of absorber, °C	115

## RESULTS OF REFINING VARIOUS OILS

Indexes	Crude deparaffinized Pennsylvania stock	Raffinate in the obtaining re- sidual oil	Extract	Desphalted con- centrate from East Texas Petroleum	Raffinate in the obtaining avia- tion oil	Deparaffinization and final earth purification of aviation oil	Crude distillation light Bibi-Eybat petroleum	Raffinate in the obtaining of distillate automobile lubricating oils	Crude deparaffinized distillate from Baku II Petroleum	Deparaffinized raffin- ate in the obtaining of distillate automobile lubricating oils	Extract
[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]	[10]	[11]	[12]
Specific gravity	0.900	0.889	1.001	0.9159	0.8778	0.8783	0.934	0.917	0.9145	0.888	-
Flash point as per Brenken, in °C	288	288	274	288	-	-	236	-	-	-	-
Viscosity, $\eta_{100}$	4.10	3.65	25	3.70	2.85	3.59	2.70	2.43	1.95	1.75	3.0
Viscosity index	96.2	101.7	-	-	-	95	32	58	40	80	-
Viscosity-weight constant -	-	-	-	0.838	0.795	0.804	-	-	-	-	-
Coke, as per Konradsen, %	1.52	0.8	-	1.43	0.2	0.26	1.043	0.19	0.35	0.18	-
Solidification point, in °C -	-	-	17.5	-	-	-17.5	-7	-3	-18	-13	-
<u>Refining procedure:</u>											
Ratio of phenol to crude stock (by weight)	1.7				3.8		1			2	
Temperature, in °C	11.0				-		70			55	
Raffinate yield, in % by weight	90				57.4		70.5			70	

[Diagram, page 71 of original]

Flowsheet for the process of oil refining with the use of  
phenol

1 - pumps; 2 - heat exchanger; 3 - steam preheater; 4 - absorber;  
5 - extraction column; 6 - phenol-water tank; 7 - condenser-cooler;  
8 - refining column; 9 - kettle; 10 phenol column; 11 - tank for  
damp phenol; 12 - vacuum ejector; 13 - steam-distillation column  
for extract; 14 - cooler; 15 - extract-heating furnace; 16 -  
raffinate-heating furnace; 17 - raffinate tank; 18 - tank for  
dewatered phenol.

THE PROCESS OF OIL DEPARAFFINIZATION WITH THE USE OF SOLVENTS  
(BENZOL AND KETONE)

One of the widespread processes for the deparaffinization of  
oils is deparaffinization with the use of a mixture of benzol, me-  
thylethyl-ketone, and toluene as a solvent.

The mixture most frequently used by the oil refineries con-  
sist of 25-50 percent dimethyketone or methyl-ethyl-ketone (MEK),  
12-25 percent toluene, and 40-60 percent benzol.

The crude stock (oil to be deparaffinized) is fed from tank  
1 by pump 2, together with a joined flow of solvent, into steam  
preheater 4, where it is heated to 60-70 °C, and is then drawn into  
water cooler 5 to be cooled down to 30-40°C. Following this, the  
solution of oil and solvent passes consecutively a "pipe-within-pipe"  
heat exchanger 6, an ammonia crystallizer 7, and follows into feeder  
tank 9. Cooled to the required (minus) temperature, the solution  
from tank 9 flows by gravity into continuously operating vacuum filter 34.

The petrolatum cake scraped off the filter is directed into a screw conveying device 33, then into tank 32, from where, via heat exchanger 20 and steam preheater 4, it is pumped into water separator 26. Into the same water separator, in the form of a condensate, enter the distilled vapors of the solvent and water from column 17, 19 and 23.

In order to extract the oil from the petrolatum cake, the latter is washed on the filter drum by the solvent, which has been cooled in ammonia cooler 31. The deparaffinized and filtered oil solution and the washing solution, being discharged from filter 34, enter vacuum collecting tank 30, from where they are fed, via heat exchanger 6, into buffer tank 10 for deparaffinized oil with solvent.

From the buffer tank, the solution is fed continuously, via steam-distillation heat exchanger 11, to the low-pressure section of evaporation column 13 for the regeneration of the solvent. In this section, 50 percent of the solvent is dissolved.

The residue from evaporating column 13 is taken in by a pump and forced, via furnace 16, into the high-pressure section of evaporator column 12. From the top of column 12, the solvent vapors pass through vertical steam-distillation heat exchanger 11 and, in the form of a condensate, flow down into collecting tank 14 for the dewatered solvent, into which tank is simultaneously fed the regenerated solvent from column 18. From collecting tank 14, the solvent is again partly taken by the pump, for mixing with fresh crude stock, into tank 35, and partly fed by pump, via cooler 31, to filter 34 for washing and extracting the oil from the filter cake. The deparaffinated oil, containing some solvent residue, follows from the high-pressure section of column 12 into steam-distillation column 17.



The solvent vapors are led off from the top of this column -- they condensate in water condenser 15 and enter water separator 26. The residue (deparaflinised oil) from column 17 is pumped, via cooler 5, into finish-oil tank 25.

The petrolatum solution, settled out from the water in water separator 26, is fed, via heat exchanger 20 and tube heater 21, into evaporation section 18 for the petrolatum. From the top of this section are led off the solvent vapors, which are condensed in water condenser 15, the condensate entering collecting tank 14. The residue from section 18 is drawn into bottom section 19 for the final steam-distillation of the petrolatum from the solvent. From the top section 19, the distilled solvent is directed into water condenser 15, and then into water separator 26. The residue (petrolatum) from the bottom of column 19 is pumped, via heat exchanger 20, into tank 24. The water solution (the sludge) is pumped into column 23, from the top of which is distilled the azeotropic mixture of the solvent and water vapors, which become condensated in water condenser 22 and enter water separator 26.

From the bottom of column 23, water is continuously discharged into the sewer system.

#### DEPARAFFINIZATION OF OIL WITH BENZOL AND KETONE

Indexes	Raffinate from Bake to petro- leum distillate	Raffinate from Baku II petro- leum concentrates	Raffinate from con- centrate of paraf- fin base Baku petroleum
[1]	[2]	[3]	[4]
<u>Composition of solvent, in percent:</u>			
Methyl-ethyl-ketone	45	45	40

[1]	[2]	[3]	[4]
Benzol + toluene	55	55	60

Ratio of solvent to crude:

Infiltration	2.5	4	4
in washing	1	1	1

## Temperature of depar-

affinization, °C	-18	-18	-24
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## Yield of deparaffined oil,

in %	78	80	75
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	Crude	Deparaf- fined	Crude	Deparaf- fined	Crude	Deparaf- fined
[1]	[2]	[3]	[4]	[5]	[6]	[7]
Specific gravity	0.886	0.900	0.905	0.914	0.808	0.903
Viscosity, ° 100	1.60	1.73	4.17	4.81	2.25	3.10
Viscosity index	110	82	100	81	110	92
Coke, as per konradsen, in %	0.10	0.15	1.35	1.6	0.45	0.6
Solidification point °C +38	-16	+45	-15	+48	-21	

[Diagram, page 71 of original]

Flowsheet for the deparaffinization of oils with the use of sol-  
vents (benzol and ketone)

1 - crude-stock tank (crude to be deparaffined); 2 - pump; 3 - water-  
vapor feed line; 4 - steam preheater; 5 - water coolers; 5 - "pipe-  
within pipe" heat exchanger; 7 - ammonia crystallizer; 8 - ammonia  
feed line; 9 - feeder tank; 10 - tank for deparaffined oil with sol-  
vent; 11 - steam-distillation heat exchangers; 12 - high-pressure  
section of evaporation column; 13 - low-pressure section of evaporation

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column; 14 - collecting tank for dewatered solvent; 15 - water condenser for solvent; 16 - tube heater for deparaffined oil with solvent; 17 - deparaffined oil distillation column; 18 - evaporation section for petrolatum, 19 - steam-distillation section for petrolatum; 20 - paraffin heat exchangers; 21 - tube heater for the pre heating of paraffin and solvent mixture; 22 - water condenser for ketone; 23 - solvent-distillation column; 24 - petrolatum tank; 25 - deparaffined oil tank; 26 - water separator for petrolatum solution; 27 - ammonia cooler; 28 - cooler for smoke gases; 29 - vacuum pump; 30 - vacuum collecting tank; 31 - ammonia cooler; 32 - petrolatum tank; 33 - screw-conveying device for petrolatum; 34 - continuous vacuum filter; 35 - tank for solvent.

#### THE PROCESS OF SECONDARY VACUUM DISTILLATION OF OILS

The essence of the process consists in the distillation of lubricating oils under a vacuum, in the presence of bleaching clay. The products of such a process are two-three types of distillate oils and residual oil.

The process is used in the refining of crude distillate or residual stock of wide fractional composition.

The crude stock (oil) is forced by pump 1 through heat exchanger 3, where it is heated to 90 - 100°C. Part (10 percent) of the total flow of the crude stock is continuously fed through mixer 7 for the intermixing of the oil and the clay.

The clay is fed from bin 4 by a belt conveyor to an elevator, which takes the clay into feeder bin 5, from where after passing automatic scale 6 it enters mixer 7. In the mixer, the oil-clay

mixture is prepared. The mixture with a temperature of 80-90°C is taken in by pump and fed into the basic flow of the preheated crude stock emerging from heat exchanger 3. The common flow then passes through heat exchanger 21, vertical tube heater 8, and, with a temperature of 360-370°C, enters vacuum column 10.

From the top of vacuum column 10, the solar fraction is taken into condenser 11, from where, upon condensating, it is pumped into a tank. Part of the solar condensate is returned to the column in the form of a spray.

The water vapor and the gases, obtained in the disintegration, enter barometric condenser 12, from where the noncondensated gases are discharged with the aid of steam ejectors 13.

The following are taken as side distillates: a light oil fraction, which is pumped, via heat exchanger 3, immersion cooler 2, into a tank, and the heavy oil fraction, which is pumped, via heat exchanger 3, immersion cooler 2, into a tank.

The liquid part of the crude stock, which entered column 10, passes through the plates located in its lower part, which serves as a contact chamber. Here, the residue is steam-distilled and stirred up by the superheated water vapor.

The contacting is controlled by an automatic oil-level regulator, with the mixture to be kept in the bottom of the column for about 20 minutes.

From the bottom of the column, the residue with a temperature of 330-340°C is taken in by pump, forced through heat exchanger 21, and, now with a temperature of 140-150°C, drawn into isolated

mixer 14. The presence of a mechanical mixer permits the continuous intermixing of the residue with the clay and prevents the settling out of the latter.

From mixer 14, the residue is fed into vacuum drum filter 15. The filter drum is covered with a monelmetal fabric, upon which is spread a layer of diatomaceous earth 75 millimeters thick. This layer of diatomaceous earth is the filtering medium retaining the clay on its surface.

The filter drum is immersed half way into the mixture to be filtered. With the rotation of the filter drum, its clean filtering surface is alternately immersed into the mixture and coming out of the mixture. Under the effect of the vacuum, the liquid mixture, passing through the filtering layer, will deposit the clay on the drum surface. With each rotation of the drum, a moving knife slices off, together with a very thin layer of diatomaceous earth, a layer of bleaching earth formed during the filtration of the oil. Since this will gradually result in that the entire filtering-medium layer will come off the drum, it becomes necessary to periodically renew the layer of diatomaceous earth upon the filter drum. In order to loosen the filtering layer, air or smoke gases are blown into the respective sections of the filter.

The filtration cycle is equal to 7 days. The filtering-layer covering operation takes 3 hours. Diatomaceous earth is mixed with gas oil and fed into the drum housing, from where, under the effect of the vacuum, it spreads around the drum surface forming the filtering-medium layer. The devitalized clay is thrown into bin 16.

From filter 15, the oil (the filtrate) is drawn into tank 17, from where it is pumped, via cooler 2, into a plant storage tank.

The vapors from tank 1<sup>7</sup> are drawn through cooler 2 and trap 18, from where they are returned to filter 15 by vacuum pump 20.

During the covering of the filter drum with a fresh filtering layer, part of the filtrate is fed into mixer 19, where the mixture of the filtrate with diatomaceous earth is prepared.

Below, there is data on the operational cycle of the installation and indexes of the yield and quality of the products obtained.

Operational cycle:

Temperature in oil-and-clay mixer, °C	80-90
Temperature of mixture as it emerges from furnace, °C	360-370
Temperature in the bottom of rectification column	330-340

APPROXIMATE MATERIAL BALANCE AND QUALITY OF PRODUCTS

Indexes	Initial	D i s t i l l a t e o i l s				Residual oil
		Solar	Light	Medium		
			automo-	automo-		
			bile lu-	bile		
			bricant	lubri-		
				cant		
[1]	[2]	[3]	[4]	[5]	[6]	
Specific gravity	0.882	0.855	0.869	0.870	0.891	
Viscosity, ° 100	1.80	1.18	1.40	1.81	4.52	
Coke, by Konradson, in % 0.1	-	-		0.05	0.3	
Acid number, in mg KOH						
per 1 gram	0.1	0.03	0.03	0.03	0.03	
Yield, in %	100.0	5.0	25	43	25	

[Diagram, page 73 of original] UNCLASSIFIED - For Official Use Only

Flowsheet for the process of secondary vacuum distillation  
of oils

1 - pumps; 2 - immersion coolers; 3 - heat exchangers; 4 - bleaching-clay bin; 5 - feeder bin; 6 - automatic scales; 7 - oil mixer; 8 - vertical cylindrical tube heater; 9 - free steam feedline; 10 - vacuum column; 11 - surface condenser; 12 - barometric condensor; 13 - steam ejectors; 14 - mixer for residue; 15 - vacuum drum filter; 16 - bin for devitalized clay; 17 - filtered oil bin; 18 - trap; 19 - mixer for the preparation of mixture with filtering agent; 20 - vacuum pump; 21 - "pipe within pipe" heat exchanger.

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